

**APPLICATION OF CHEMOMETRICS TECHNIQUE TO NUTRITIONAL
QUALITY EVALUATION OF SELECTED UNPROCESSED AND PROCESSED
WHEAT PRODUCTS IN MINNA, NIGER STATE**

BY

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ABSTRACT

The study focused on the application of chemometric technique to nutritional quality evaluation of selected processed and unprocessed wheat products in Minna, Niger state. Fourteen wheat samples were collected from three markets in Minna. Proximate compositions of the samples were determined using standard methods. The samples were first wet ashed using H_2SO_4 , H_2O_2 and deionized water before instrumental analysis using FES for (Na, K, Mg and Ca), colorimetry (P) and AAS for (Fe and Zn). The proximate compositions indicated moisture contents range between 8.83-13.74% and 7.7-11.00% for the processed and unprocessed samples respectively. The unprocessed samples had more ash, carbohydrate, fat, fibre and energy values of 1.44-1.89, 66.39-70.84, 5.59-6.92, 1.16-1.01% and 369.71-383.95 kCal/100 g respectively. The protein contents ranged from 12.25-16.61 % for all the samples. The mineral contents (mg/100g) were: Na (5.91-6.82 and 3.20-4.61), K (506.33-558.35 and 161.57-482.50), Ca (50.24-56.20 and 27.43-54.40), Mg (68.64-92.23 and 46.17-66.30), P (156.12-278.32 and 128.23-268.30), Fe (4.22-5.18 and 2.87-6.58) and Zn (5.19-6.43 and 2.57-5.24) for the unprocessed and processed samples respectively. The unprocessed samples had more amounts of K, Zn, Na, Ca and Mg while the processed samples had more Fe and P contents. Physical tests (farinography and sedimentation analysis) suggested that the processed samples had better physical properties such as rheological and baking properties and better gluten contents. PCA and HCA revealed that substitutability is peculiar to either the processed or unprocessed samples. Correlation matrix further revealed that there was loss of identity due to processing as the correlation observed in the unprocessed samples could not correlate with the processed samples. This study shows that the unprocessed samples are richer in both micro and macro nutrients and therefore, could be suggested for more consumption than the processed.

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LIST OF ABBREVIATIONS

AAS	Atomic absorption spectrophotometer
FES	Flame Emission Spectroscopy
DRI	Dietary Reference Intake
RDA	Recommended Dietary Allowance
DDT	Dough Development Time
TBD	Time To Breakdown
FQN	Farinograph Quality number
WAC	Water Absorption Capacity
PTH	Parathyroid Hormone
GPF	General Purpose Flour
WH	Whole Wheat
PC	Principal Components
ATP	Adenosine Triphosphate
PCA	Principal Component Analysis
NSP	Non-Starch Polysaccharide
PLS	Partial Least Squares
MATLAB	Matrix Laboratory
ICPMS	Inductively-Coupled Plasma Spectroscopy
GMP	Good Manufacturing Practice
FTIR	Fourier Transformed Infrared
BU	Berbender Unit
HCA	Hierarchical Cluster Analysis

CHAPTER ONE

1.0

INTRODUCTION

1.1 Background to the Study

Food is the most essential of all the basic needs of every living thing. It provides nutritional support when consumed (through eating or drinking) and thus, help to maintain life and growth. It is commonly obtained from plant (crops, and herbs) and animals (meat, fish, milk) (Ward *et al.*, 2008). Usually, food is known to constitute important nutrients such as fats and oils, proteins, carbohydrates, minerals and vitamins which when consumed, is absorbed by the body cells to give energy and maintain or promote growth (Gupta *et al.*, 2010).

Food rich in protein such as meat, egg and milk help to build, repair and maintain muscles, blood, skin, bones and other tissues in the body. Fats serve as body's secondary source of energy than other nutrients, though they are difficult to burn. Carbohydrates can be subdivided into starch and sugar thus, serves as the body's main source of energy. Starch-rich foods are wheat, maize, potatoes while sugar-rich foods are fruits, honey, and other sweetened foods (Brody, 2014).

Vitamins and minerals are required in minute quantities and hence are referred to as micro nutrients. They are essential for good health as they regulate or control many metabolic processes (vitamins). Minerals are essential chemical elements present in food. They are required to aid proper functioning of the body metabolic activities and also build or repair body tissues such as bones. The major mineral elements are sodium, potassium, calcium, phosphorus and magnesium (Vasfiye *et al.*, 2015).

Wheat (*Triticum*) which are cereal-based foods like rice, millet and guinea corn are cultivated because of the edible component of their grains. Wheat is made up of germ, bran and endosperm. Wheat is highly nutritious and it is a rich source of carbohydrates, minerals, fats, vitamins and proteins (macronutrients). Wheat is also known to be rich in mineral elements such as potassium, phosphorus and zinc (Ward *et al.*, 2008).

Wheat is one of the major cereal crops grown in Nigeria, about 60,000 tonnes is harvested annually (Oladumoye *et al.*, 2010). All over the country, natural wheat (whole-wheat meal) and commercial products are being consumed unavoidably on daily basis in the form of bread, cake, biscuit, wheat-meal and so on. This conferred on it an important position in the nutritional ranking as it is known to contain high starch, rich dietary fibre, nutritious protein and lipids and also rich in important fatty acids (Saka and Lawal, 2009).

It is also a good source of micro nutrients such as vitamins. It has high yield of gluten fraction (a polymer which improves the rheological properties of wheat flours), thereby enhancing its visco-elastic properties (the viscous and elastic property of a material when undergoing deformation) and thus, allows its dough to be processed into bread, pasta, noodles and other food products for both human and livestock feeds (Shewry, 2009).

Nutritional parameters on wheat include farinography, sedimentation, proximate composition as well as parameters such as vitamin A, maltose, minerals and protein. Thus, its nutritional compositions are evaluated and ascertained to be in conformity with the required standard. Having known the nutritional quality of product(s), quality control and assurance practice help consumers of such product(s) to make knowledgeable choices as

manufacturers are required to adhere to the specified standards established by standard organizations (Udeme *et al.*, 2014).

Also, nutritional information about these products enhances fair competition between manufacturers and help in ensuring that manufacturers keep to the standard specified by the government and other relevant authorities thereby minimizing economic fraud (Asamudo *et al.*, 2017).

Quality can be defined as fitness for purpose, it determines the ability of a product to possess properties that can make it suitable to meet the expressed or potential needs of its users. Authenticity deals with moves made at ascertaining the quality of nutrients or compositions said to make up a food substance according to its nutritional label. Thus, enabling quality assurance of such food product and discouraging economic fraud (Lia *et al.*, 2010). Chemometrics is a statistical analytical technique used to derive information from results obtained or measurement(s) made on chemical systems. It is a scientific approach which enables the analysis of multidimensional data using mathematical statistics, probability theory and information technology (Yu, 1992; Mike, 2016).

Unlike traditional statistical methods which are often inadequate for a more accurate and deeper interpretation of results as it allows valuable information to be obtained from a wide range of complex data sets and facilitates the detection of hidden relationships between variables. (Kamal and Karoui, 2015). Examples of these techniques are; principal component analysis (PCA), hierarchical cluster analysis (HCA), cluster analysis (CA), linear discriminant analysis (LDA) and Fischer-Routy discriminant analysis (FDA). The principal component analysis (PCA) is commonly employed in exploratory data analysis as it shows the original measurement by discovering the dominant factors and keeping the

interference factors at minimum significance (Liang *et al.*, 2020). Hierarchical cluster analysis (HCA), this is most advantageous in its flexibility to alter the similarity measurement criteria and the applied linkage method to suit different uses. Cluster Analysis (CA), this is employed for easy classification of objects based on their quantitative characteristics (Huang *et al.*, 2014). Linear Discriminant Analysis (LDA) and Fisher-Routy discriminant analysis (FDA) are both discriminant analytical technique that assign samples to specific classes. It shows linear classification or aids dimensionality reduction and are used to set up a discriminate function (Chen *et al.*, 2012).

Balanced diet is vital in maintaining good health. Thus, the nutritional quality of foods is an important aspect that should be vigorously considered especially with respect to metal intake such as iron, calcium, magnesium, potassium, sodium, selenium, manganese, copper, chromium and zinc (Ward *et al.*, 2008). Minerals are inorganic elements, usually required in trace amounts from less than 1 to 2500 mg per day, depending on the mineral. They are present in body tissues and fluids for the maintenance, repair and proper functioning of certain physicochemical processes which are important to life. Minerals are classified into two groups Essential and Trace minerals, depending on the quantity required and found in the body, essential minerals are present in the largest amounts, they are the macro elements which include calcium, magnesium, potassium and sodium (Udeme, 2014).

Wheat is generally not classified by variety. Instead, it is classified based on the time of year the wheat is grown and the milling and baking quality of the flour produced. Within each class there is a group of different varieties of wheat with similar characteristics. Most of the wheat produced is used for human consumption and a large range of ingredients and foods are produced from it because of its unique properties (Ward *et al.*, 2008).

Atomic absorption spectrometry (AAS) is an analytical technique that measures the concentrations of elements. Atomic absorption spectrophotometer is so sensitive that it can measure down to parts per million of a gram in a sample. The technique makes use of the wavelengths of light specifically absorbed by an element. They correspond to the energies needed to promote electrons from one energy level to another (higher energy level). Atomic absorption spectrometry has many uses in different areas of chemistry such as clinical analysis, environmental analysis, pharmaceuticals, industries and mining (Wiley *et al.*, 2015).

The technique makes use of absorption spectroscopy to assess the concentration of an analyte in a sample. It requires standards with known analyte content to establish the relation between the measured absorbance and the analyte concentration (Adebiyi *et al.*, 2017).

1.2 Statement of the Research Problem

Lack of detailed nutritional information on wheat products had made some consumers victims of economic and food fraud as some wheat flour and product may fall below the standard quality composition contrary to their nutritional labeling. Thus, hindering the consumers from making informed choices. Several conventional statistical analytical techniques, have proven inefficient in the proper interpretation of analytical data as they are unable to give detailed information on principal components, clustering, similarities and hidden relationships existing amidst the chemical data set.

1.3 Justification of the Study

Quality evaluation of nutritional parameters of selected unprocessed and processed wheat products will give better information of the nutritional composition of the wheat products.

The application of chemometric technique on the data obtained from proximate composition, mineral content and physical tests (farinography and sedimentation analysis) will reveal more information about the wheat products.

1.4 Aim and Objectives of the Study

The aim of this research was to apply chemometric technique to interpret the nutritional parameters of selected unprocessed and processed wheat products in Minna, Niger State.

The above aim shall be achieved by the following specific objectives;

- i. Determination of the proximate composition of unprocessed and processed wheat products using standard methods.
- ii. Quantification of the mineral content (Na, K, P, Ca, Fe, Mg and Zn) of the unprocessed and processed wheat products using atomic absorption spectrophotometry, flame photometry and colorimetry.
- iii. Evaluation of the physical properties through farinography and sedimentation analysis.
- iv. Performance of principal component, correlation and hierarchical cluster analysis on the chemical data for pattern recognition and characterization.

CHAPTER TWO

2.0

LITERATURE REVIEW

2.1 Wheat

Wheat (*Triticum aestivum*L.) are cereal-based foods and it is counted among the big three cereal crops with more than 600 million tons harvested annually. It is the dominant crop in temperate regions where it serves as food for both human and livestock consumption. Its success partly depends on its adaptability and high yield but also on its gluten-protein-fraction which gives it the viscoelasticity of its dough and hence, allows it to be processed into noodles, bread, pasta and other wheat food products. It is one of the most important crops for the majority of world's populations. It is a vital staple food of about two billion people (36% of the world population).

Worldwide, wheat provides nearly 55% of the carbohydrates and 20% of the food calories consumed globally (Shewry, 2009). Averagely, it supersedes in production and consumption every other grain crop (including rice, maize, etc.) and thus, the most vital cereal crop of the world, which is cultivated over a wide range of climatic conditions and the understanding of genetics and genome organization using molecular markers is of great value for genetic and plant breeding purposes.

Wheat (*Triticum aestivum*L.) and other major crop plants such as wheat (*Triticum aestivum*L.), barley (*Hordeum vulgare* L.), oat (*Avena sativa* L.), rye (*Secale cereale*L.), maize (*Zea mays* L.) and rice (*Oryza sativa* L.) belongs to the grass family *Poaceae* (*Gramineae*). *Triticeae* is one of the tribes containing more than 15 genera and 300 species including wheat and barley. Wheat belongs to the tribe *Triticeae* (*Hordeae*) in the grass family *Poaceae* (*Gramineae*) (Goncharov, 2011). Among the several flowered spikelets, wheat plants are sessile and alternate on opposite sides of the rachis forming a true spike.

Wheats (*Triticum*) and ryes (*Secale*) together with *Aegilops*, *Agropyron*, *Eremopyron* and *Haynalidia* form the subtribe *Triticineae* (Knott, 2012).

Wheat was first classified by Linnaeus in 1753. The chromosome number sets (genomes) for each commonly recognized type was reported by (Tsunewaki, 2016). This was a turning point in *Triticum* classification. It separated wheat into three groups. Diploids had 14 ($n=7$), tetraploids had 28 ($n=14$) and the hexaploids had 42 ($n=21$) chromosomes. Bread wheat is *Triticum aestivum*. *T. durum* and *T. compactum* are the other major species. All three are products of natural hybridization among ancestrals no longer grown commercially (Shewry, 2009).

In Nigeria, common wheat (*triticum aestivum*) is one of the major cereal foods grown. About 60,000 tonnes is harvested annually (Saka and Lawal 2009). A wheat grain constitutes a bran, germ and an endosperm. Natural and commercially processed wheat foods are unavoidably consumed on the daily basis in the forms bread, pasta, wheat-meal (tuwon-alkama), semovita and so on. They are nutritious and rich in high dietary fibre, protein, certain fatty acids and micronutrients (such as vitamins) which are particularly enriched in whole grain products. However, it is predicted to cause a number of reactions in humans including intolerance (known as celiac disease). (Garcia-Alvarez *et al.*, 2011).

2.1.1 Reviews on the physicochemical studies of wheat

Balarabe *et al.* (2017) carried out physicochemical and sensory evaluation of bread produced from various indigenous yeast isolates so as to give a deeper understanding concerning the overall quality of bread. Four different species of yeast isolated from pineapple, palm wine and sweet orange were characterized and used to ferment wheat flour dough. Certain bread samples were seen to have high specific volume while some were low depending on the nature of yeast employed. The proximate composition of each bread

sample evaluated revealed the following; moisture content ranged from (0.2959-24.1), protein (3.325-5.425), fat (4.165-6.80), fibre (1.49-6.50), ash (1.331-3.103), carbohydrate (43.325-89.112) and energy (256.20 - 407.2298). The bread sample PW7B had the highest value of moisture (24.10), protein (4.90) and fat (7.45) while AC1B had the highest value of carbohydrate (89.11) and energy (407.23) (Ambi *et al.*, 2017).

Samuel (2016), assessed the quality and sensory properties of sorghum (*sorghum vulgar*)-wheat (*Triticum aestivum* L.) flour cookies by starting their physicochemical parameters and sensory parameters and was compared with that of a controlled cookie from wheat flour. Sorghum was produced from whole grains and the. Incorporated at 5-50% level in wheat flour. There was an observed increase in the proximate composition (moisture, ash, crude fibre, protein, and fat contents) with corresponding decrease in the starch and sugar contents as the percentage substitution increased. The proximate compositions (moisture, ash, crude fibre, fat, protein, starch and sugar contents) of wheat flour are 8.64 ± 0.18 , 1.5 ± 0.04 , 1.42 ± 0.05 , 2.29 ± 0.06 , 8.48 ± 0.12 , 76.92 ± 0.31 and 1.68 ± 0.13 , while the proximate composition (moisture, ash, crude fibre, protein, and fat contents) of sorghum flour were 10.28 ± 0.39 , 2.41 ± 0.49 , 2.32 ± 0.14 , 3.83 ± 0.21 , 10.72 ± 0.24 , 70.38 ± 0.10 and 1.16 ± 0.02 . The proximate composition of composite flour with different level of Sorghum was also obtained.

Goshal *et al.* (2016), studied the effect of partially purified xylanase on quality attribute of whole wheat bread during storage. It was observed that the xylanase incorporated bread gained an improved storage quality for example, reduced water absorption for dough preparation, reduce moisture-loss from bread during storage, increased specific volume, increased shelf life and low firmness.

Wheat-cassava and wheat-potato composite flour for the production of bread were studied when the optimization, physicochemical and thermal evaluation were carried out. The two species of cassava and potato were processed into flour and used as substitute at varying ratios for wheat flour. These substitutes were used to produce bread, the proximate and hydrogen cyanide content was investigated. The results from the proximate composition of the composite flour, that is, wheat-Irish potato (WIP) and wheat-red potato (WRP) in the order of moisture, ash, crude fibre, crude fat, crude protein and carbohydrate are 8.56 ± 0.08 , 2.62 ± 0.03 , 1.68 ± 0.04 , 2.82 ± 0.10 , 12.21 ± 0.01 and 72.11 ± 0.30 . While (WRP) in the same order as above are 5.86 ± 0.40 , 1.70 ± 0.10 , 1.72 ± 0.02 , 2.45 ± 0.04 , 11.22 ± 0.60 , and 77.06 ± 0.60 . The mineral analysis of WIR were also reported in the order Na, K, P, Zn, Fe, Mg and Ca as follows: 450.38 ± 0.50 , 893.06 ± 0.08 , 39.86 ± 0.06 , 7.67 ± 0.20 , 9.76 ± 0.20 , 173.50 ± 0.10 and 25.01 ± 0.10 while WRP in the same order are as follows: 486.05 ± 0.70 , 782.30 ± 4.20 , 90.18 ± 0.03 , 7.89 ± 0.10 , 5.78 ± 0.03 , 4.53 ± 0.040 and 32.82 ± 0.20 (Arewa, 2017).

In order to devise a strategy to curb protein malnutrition through composite flour technology, wheat variety and mung bean variety (NM-2006) were used to prepare flour blends and were investigated for their application in the baking industry. Farinographic results indicating 60.8% water absorption capacity and mixing tolerance index of 120bu were found to be higher in 15% and 20% mug bean flour blend respectively. Also, the presence of mung bean led to the increase in protein content from 5.40-9.30%, fat 21.30-23.70% and fiber 0.40-0.95%. Interestingly, the calorific value also increased from 485-501.10Kcal/100g. The evaluation of the mineral content shows that there was tendency of increase for sodium, potassium, iron, magnesium, zinc and manganese as mung bean flour

increased gradually. The sensory attribute of the baked product also showed a great improvement (Imran *et al.*, 2017).

Also, Oladumoye *et al.* (2010), carried out the physicochemical characterization of flour as they affect flour quality and their subsequent products. This involves comparative evaluation of particle size, moisture content, water absorption capacity, pasting viscosity, fat, protein, bulk density and color. Of wheat, cassava, maize and cowpea flours using standard methods. The breads produced from composite flours were in ratio. 50:30:20, 60:20:20, 70:20:10, 80:10:10, 85:10:5 and 90:5:5 of wheat-cassava/maize-cowpea flours respectively. The sensory and proximate analysis of the products were carried out and their results are in the order of particle size, moisture content, bulk density, water absorption capacity, fat and protein contents of wheat, cassava, maize and cowpea flours are as follows: 154-343 μm , 13.3-14.9% db, 327.4-497.5 kg/m^3 , 31.9-221.8%, 1.01-2.3% and 2.6-19.39%. Wheat flour was observed to have the lowest pasting temperatures of 56.1 $^{\circ}\text{C}$. Significant difference at $P < 0.05$ were recorded between most of the properties of the flour composite bread of 85% wheat, 10% cassava, 5% cowpea, 90%wheat, 5% cassava, 5% cowpea and 90% wheat, 5% cowpea were accepted by sensory panelist and substitution with cowpea fruit, improved the protein content of the bread.

Diosi *et al.* (2015) evaluated the role of farinography and sedimentation properties on wheat qualities of twelve competitive wheat flour samples in Hungary. Their farinography properties in the following order; water absorption capacity (%), dough development time (mm:ss), stability (mm:ss), mixing tolerance index (BU), dough consistency (BU) and farinography quality numbers were reported as follows: 59.6-63.5, 1:07-3:08, 1:21-1:06, 18-35, 485-498 and 30-36 respectively. The range of their sedimentation volumes were also reported to be between 32 and 35 cm^3

Ali *et al.* (2015) determined the effect of carbohydrase on dough rheology and end quality of cookies. This was done by adding carbohydrase in wheat flour samples and then measuring their sedimentation and farinography properties alongside the control samples respectively. The range of the farinograph results of the carbohydrase-induced flour samples and the control flour samples were reported in the following order; water absorption capacity (%), dough development time (mm:ss), stability (mm:ss), mixing tolerance index (BU), dough consistency (BU) and farinography quality number were reported as follows: 55-59, 4:00-6:35, 1:50-5:08, 28-45, 490-495 and 35-48 respectively for controlled samples and 56-60, 1:00-1:36, 1:55-5:00, 16-22, 492-498 and 23-33 respectively. Their sedimentation values were also reported to range from 29-33cm³ (control samples) and 31-35cm³ (carbohydrase-induced samples).

Ahmed *et al.* (2015) evaluated the rheological and physicochemical properties of soft wheat flours obtained from three different countries (Pakistan, Ukraine and India). The research involved the sedimentation analysis, starch damage test and farinography test. The farinograph result were reported in the following order; water absorption capacity (%), dough development time (mm:ss), stability (mm:ss), mixing tolerance index (BU), dough consistency (BU) and farinography quality number were reported as follows: 56.48-57.33, 2:07-2:45, 2:16-3:05, 44.60-48.55, 475-486 and 46-53 (Pakistan), 57.99-62.23, 2:50-4:16, 2:00-3:13, 23-34, 489-493 (Ukraine) and 42-58 and 63.62-65.66, 1:36-1:45, 2:19-2:45, 38-47, 479-493 and 37-43 (India) respectively. The range of their sedimentation results were reported in the order; Pakistan, Ukraine and India (27-35cm³, 2428cm³ and 31-34cm³) respectively.

Zuzana *et al.* (2017) reviewed the chemical composition and nutritional quality of wheat grain and found out that the deficiencies in wheat food micronutrients such as iron, zinc

and vitamin A (hidden hunger) affects over there billion people worldwide. This this encourages consumers to make informed choices of foods of high nutritional value and health beneficial compounds. In order to ensure balanced nutrient supplies in foods, food systems must be modified to encourage continuous availability and quality in both adequate and cost-effective amounts.

Roy *et al.* (2015), evaluated the mineral composition of wheat flour in the cities of Brazil. Elements such as Ca, Cu, Mg, Mn, Fe, P, K and Zn weekend evaluate by first collecting the samples appropriately and subjected to digestion using nitric acid and hydrogen peroxide unanswared open system. Inductively coupled plasma optical emission spectroscopy (ICP-OES) was the instrumental technique employed. After the analysis, it was observed that the macro nutrients; Ca, Mg, K and P have average contents of 0.27, 0.35, 1.71 and 0.81-7.15mg/g respectively. While micronutrients; Cu, Fe, Mn and Zn have Average contents of 1.84, 37.8, 8.20 and 9.4ūg/g for concentration ranging from 1.00-2.80, 10.5-146.6, 3.9-14.7 and 5.1-13.9ug/g respectively.

2.1.2 Benefits of wheat

Wheat is the foundation of many wholesome and healthful products enjoyed across the globe. It provides energy through the complex carbohydrates it contains, rich source of fiber, magnesium, B-vitamins, Felix acid phytochemicals. All these aforementioned nutrients contribute immensely to the health and dietary benefits of wheat foods as it helps to prevent one of the diseases plaguing the world today for example; diabetes, some cancers and neural tube birth defects (Shewry, 2009).

The nutrient richness of wheat foods make it fit consistently for the preparation of different dishes and desserts. The nutritional components of its bran, germ and endosperm have encouraged its drastic increase in consumption as either a whole-wheat-meal or their

processed foods. It is a major source of vegetable protein in food than any other cereal food. Also, when compared to other cereal crops, it is more adaptable to a wide range of climate or growth conditions. Thus, enhancing its possibility of cultivation in different parts of the world and hence, makes it globally available. The taste of its products through sensory evaluation, the price of both locally and commercially produced commodities, its availability and nutritive property also affects its preferential choice of consumer. Thus, helping it to meet the consumer required properties which make it a major staple cereal food globally (Gayathri and Rashmi, 2017).

Wheat bran also helps in producing short chain fatty acids which improves gut-health as the carbohydrate content undergo fermentation. The short chain fatty acids are known to nourish concocytes and decreases intestinal pH which enhances the growth of important lactic acid bacteria. Feruloyl oligosaccharides (phenolic compounds) found unquestionable bran protects against the occurrence of the free radical-induced oxidative damage in human white blood cells (erythrocytes) (Mohindra *et al.*, 2002).

Consumption of wheat (especially whole wheat) foods aids better frugal bulking and delays gastric emptying. It has also been discovered to offer protection against colon cancer and found to be better pectin as diluter in animal models. Thus, diluting potential carcinogens and their promoters and also prevents/reduces their access to colon-cell lining (Zuzana *et al.*, 2017).

2.1.3 Quality measurement

The need for quality measurement especially in the processing of food cannot be over emphasized, as quality. Quality is defined as "fitness for purpose ". Thus, it is of paramount importance/need to determine/measure the quality criteria of a substance in order to checkmate/ascertain its fitness for its envisaged purpose (s). Various methods are being

adopted all over the world for measure determine quality, this is influenced by factors such as mature, state, composition, geographical location, availability of equipment/technology and more. These factors vary from place to place and are efficiently utilized to ensure product quality and hence, it's purpose (Chiarini, 2017).

Quality measurement in food processing not only ensures food quality, it also enhances food safety by limiting or preventing fraudulent act by manufacturers through false nutritional labeling and food contamination that may arise through the use of harmful additives, and improper handling. Measurement of food quality will help to ascertain if constituents used are in agreement with specified regulations by relevant authorities/bodies.

Quality assessment aids or encourages proper food processing ethics, from the selection of raw materials to be used, processing techniques employed, packaging and the distribution of finished products. Thereby, enhancing a finished product that is safe, legal and meets the consumer demand as well as the regulatory standard (Oscar *et al.*, 2017).

Quality control/assurance can be enhanced through the use of accurate and repeatable measurement procedures, this helps to minimize/erase errors occurring during formulation and detect/trace non-conformity in food processing. Several processing methods or techniques have been improvised to enhance improved microbial safety and nutritional quality which modifies psychochemical properties, increase production and process efficiency (Thapa, 2013).

Physical cleaning of raw materials and processing equipment so as to adjust the raw materials to appropriate moisture content and mechanical reduction to desired particle size and minimize contamination of constitutional make-up to enhanced product quality as a result of followership of the good manufacturing practice (GMP rules). Thereby easing the process of quality control and assurance (Alldrick, 2010).

2.1.4 Review on application of chemometrics to food quality evaluation

Forina *et al.* (2018), Reported that chemometrics was defined by William Gosset as a science of multivariate statistics. This was later defined to mean the chemical discipline which uses mathematical, statistical and probability methods to predict or select an optimal measurement procedures and experiments to provide maximum chemical information by analyzing chemical data (Reiner *et al.*, 2013).

The desired objectives and possible improvement during the analysis of a chemical data can be achieved by starting with a clear definition of the problem statement and objectives, the chemical system associated with it, practical constraints and proper collection/treatment of sample and selection of analytical techniques. Error or failure in any of the aforementioned steps above no longer guarantee accurate results even with the potential use of chemometric tool (Michelle *et al.*, 2009).

In recent times, the application of chemometrics to food quality assessment/measurement has been one of the most reliable ways through which an in-depth knowledge of the constitutional make-up of food is obtained. Unlike other conventional statistical analytical techniques, it consists of multivariate analytical tools that are capable of revealing hidden-relationship/information regarding the data obtained from chemical systems. In the areas of food authenticity /food quality assessment, chemometric tools in conjunction with various analytical techniques such as HPLC, FTIR, MIR among others play vital role in analyzing, differentiating, distinguishing and revealing vital similarities between food quality parameters as it reduces calibration challenges for analysis of spectral data, encourages

improved method development and employs routine use of statistical models for data analysis (Semih and Vasfiyen, 2015).

Problems resulting from food chemistry and the relationship between food quality parameters such as consumer preference, food processing and storage, method of production, physical characteristics and sensory evaluation require the use of chemometric tool/technique and strategies (Martens and Russwurm, 2016).

Since the origin of chemometrics, chemometricians have done a great deal in the study of problems relating to food quality and authenticity which makes it one of its largest field of applications. Data obtained from chemical systems are often used to build and checkmate new chemometric techniques and strategies. Food chemistry or composition of every food varies and sometimes complex depending on the nature of raw materials used, preparatory procedures, storage/ripening condition employed and so on. Thus, information on both physical and chemical properties can be obtained and are often important in the description of a food. The physical and chemical properties hence provide the basis for its classification and nutritional labelling. Since food composition is important/fundamental to solve problems of quality control, assurance and classification, due to its possible complexity, the use of chemometrics is frequent and in the determination of chemical information through multivariate calibration; to build models for control objectives and identification, to study food property relationships and how it can be optimized, to study the relationships between composition and biological/physical behavior. (Michelle *et al.*, 2009).

Guidetti *et al.* (2010), assessed Vis/NIR device (450-980nm) combined with hyperspectral images in order to predict the ripening indexes (firmness and soluble solid content) and the presence of compounds having functional properties (anthocyanins, flavonoids, ascorbic

acid and polyphenols). A very good predictive statistics were observed using correlation coefficient (r) between 0.80 and 0.92 for the regression models of homogenized samples having (r) greater than 0.8 for all indexes.

De-Temmerman *et al.* (2014) proposed NIR (near-infrared) reflectance spectroscopy for the assessment of in-line moisture content present in semolina pasta immediately after extrusion process. Extrusion of several pasta samples with different moisture concentration were carried out and reflectance spectra between 308 and 1704nm were measured. An appropriate prediction model was adopted based on the PLS (partial least square) methods using leave-one-out cross validation. Very good results were obtained with $R^2= 0.956$ and a very low level RMSECV was observed. Thus, enhancing the measurement of the moisture content with low-cost sensor.

The predictions of nutrients in a wide range of bread varieties produce mainly of wheat and rye origin were assessed. Very good results were observed using near infrared spectroscopy coupled with a chemometric tool (Principal Component Analysis) PCA. This was used to determine the total content of carbohydrate and energy from NIR days with R^2 values of 0.98 and 0.99 respectively (Sørensen, 2009).

Jia *et al.* (2017), carried out the rapid determination of farinograph parameters of wheat flour using data fusion and forward interval variable selection algorithm. In this experiment, he was able to detect their water absorption (WA), dough development time (DDT), dough stability (DS) and degree of softening. (DOS). Although, these analyses are labor-intensive and time consuming. Success was achieved by the combination of NIR and MIR regions to predict the flour farinograph parameters.

Haseeb *et al.* (2016), implemented process analytical technologies to predict analytical, rheological and baking parameters of wheat flours using fluorescence spectroscopy and

partial least square regression models coupled with genetic algorithm. These chemometric tools were applied on spectral data to optimize the prediction of these quality parameters mentioned earlier above. The linear regression models obtained for protein, wet gluten and sedimentation value of group R² of 0.95 and 0.77 respectively were obtained settling at 0.78 passing temperature. Other parameters like bread volume and moisture were determined with good accuracy revealing R² of 0.86 and 0.95 respectively.

Adebiyi *et al.* (2017) performed chemometrics on the chemical data obtained from the proximate compositions and mineral contents of cereal grains. The grains were milled following standard procedures and their proximate compositions and mineral contents were evaluated. PCA was carried out the data sets and the following information were revealed; A cumulative variance capture of 32.13% (PC2) and 66.00%(PC1) was observed for the samples. The loading plot revealed crude fibre and Ca (Calcium) as the major principal components in the proximate compositions and mineral components respectively. The biplot also revealed that none of the sample is characterized by the principal components.

Wang *et al.* (2020) carried out HCA and PCA on the proximate composition and mineral components of wheat flour varieties. The PCA captured revelations from Eigen value plots, loading plots and biplots. The eigen value plot revealed a cumulative variance capture at 21.25% (PC2) and 69.33% (PC1), the loading plot revealed K (potassium), Na (Sodium) and crude protein as the major principal components the proximate composition and mineral contents respectively. The biplot indicated that all the samples are characterized of the principal components. The HCA dendrogram showed that majority of the samples are substitutable.

Kibar (2019) assessed the mineral and morpho-physiological properties of de-hulled eikorn wheat samples during storage at varying moisture contents. PCA, HCA and correlation

analysis were performed on the data-sets obtained from the proximate compositions and mineral contents of the wheat samples. Significant correlations were observed between ash and K (potassium) and protein and pH. Negative correlations were also observed between P (phosphorus), Na (sodium), Ca (calcium), Mn (manganese), Co (cobalt) and Cr (chromium) when compared to K. The PCA loading plot revealed K, P, Ca, fiber and carbohydrate as the major principal components in the samples at cumulative variance capture at 54.58% (PC2) and 35.72% (PC1) for the proximate and mineral content data sets, the biplot revealed that samples with low moisture are more characterized by the principal components than those with high moisture contents. The HCA dendrogram grouped the samples into micronutrient-rich and less micronutrient-rich samples (mineral contents) and macronutrient-rich and less macronutrient-rich samples (proximate composition).

2.2 Proximate Composition

The term proximate analysis implies the examination of biological material as a decomposition of human consumable food into its major constituents. It gives the potent approximation and easy verification of nutritional components of packaged consumable goods (Onuwka and Onwuka, 2005). All proximate parameters were extracted from Association of Analytical Chemist (AOAC), methods of analysis. (AOAC, 2006)

2.2.1 Moisture content

This is known as weight loss experienced after drying a known weight of food at constant (John *et al.*, 2017). This technique is employed for most foods though significant losses of volatile materials may occur. The total solid or moisture content of food is important in the determination of food quality, Shelf life and its resistance to spoilage. Assessment of the total solid is also important as it helps to estimate the amount of food ingredients on a dry weight basis. The dry content/matter remaining after drying is usually termed as total

solids. The moisture content of a food substance must be estimated so as to give the proportion of carbohydrate present. This analysis can be carried out through various methods; each method presents its own level of precision and accuracy as well as its merits and demerits. Thus, all experiment is advised to be carried out at least three times to obtain an average value at the end of the experiment.

2.2.2 Ash content

Total ash is the inorganic residual remaining after combustion in a muffle furnace. This reflects the quantity of mineral matter present in the flour (Sobota *et al.*, 2020). Acid insoluble ash reflects added mineral matter in milled products such as dirt, sand, and so on. The non-combustible ash is then analyzed to estimate the elemental composition of the sample and the presence of impurities. Ashing methods include; dry ashing, wet ashing and sulfated ashing. This is done by burning a known weight of food substance at 550°C until all carbon content has been removed. The inorganic constituent matter of the food is then obtained as ash or residue. Though, the residue may contain Sulphur and phosphorus from protein (from organic origin). Some volatile materials such as sodium, chloride, Sulphur and phosphorus may be lost during ignition. The residue obtained is not a true representative of the inorganic constituents in the food both qualitatively and quantitatively. However, information regarding the rate of mineral concentration in the food is obtained. A standard approach to the assessment of as content food sample is dry again. In this case, the food sample is ignited between 550-600°C and all the organic materials are oxidized without flaming. The ash/residue is the inorganic particles remaining that are not volatilized at the temperature, it is determined from weight loss during complete oxidation. In the case if elemental analysis, wet ashing is employed. Concentrated perchloric acid and nitric acid or sulphuric acid and nitric acids are used to oxidize the organic content of the

food sample. The soluble minerals are dissolved in the nitric acids since the acids are partially removed by volatilization (AOAC,2006).

2.2.3 Crude fat

This is referred to as the ether extract and it is determined by subjecting the food a continuous extraction with Perth petroleum ether for a particular period. The residue, remaining after evaporation of the solvent used is known as the ether extract. It contains alcohol, organic acids, lipids and pigments. Currently, before the extraction with ether, the sample is first hydrolyzed with sulphuric acid and the residue resulting from this process is the acid ether extract. Solvents such as carbon tetrachloride, chloroform, carbon disulphide and petroleum distillates of higher or lower boiling points, dissolve fats and oils though the result and the composition of the extract differ somewhat with the solvent. Less polar solvents such as petroleum ether and diethyl ether can be used to extract free fat unlike the bound fat, which requires more polar solvents for their extraction. Thus, the amount of fat extracted from food products depends on the technique employed (Samuel, 2016).

2.2.4 Crude protein

The crude protein (CP) content is assessed from the amount of nitrogen in the food, determined by a modification of a technique originally devised by Kjeldahl over 100 years ago. In this method, the food is digested with sulphuric acid, which converts all the nitrogen present to ammonia except those in the form nitrate and nitrite. This ammonia is liberated by adding sodium hydroxide to the digest, distilled off and collected in a standard acid, the quantity collected is determined by titration or by an automated colorimetric method. It is assumed that the nitrogen is gotten from protein containing 16% nitrogen and by multiplying the nitrogen figure by 6.25 ($100/16$), an approximate protein value is

obtained. This is not ‘true protein’ since the method determines nitrogen from sources other than protein, such as free amino acids, amines and nucleic acids and the fraction is therefore designated crude protein (Goshal *et al.*, 2016).

2.2.5 Carbohydrate

The carbohydrate of food is contained in two fractions, the crude fibre (CF) and the nitrogen-free extractives (NFE). The crude fiber is determined by subjecting the residual food from ether extraction to successive treatment with boiling acid and alkali of defined concentration, the organic residue is the crude fibre. The amount of carbohydrate present is the sum of the amount of moisture, ash crude protein, fat and crude fibre subtracted from 100 (Salau, 2012).

2.2.6 Crude fibre

Lignocellulosics are the major component of fiber, that is, lignin, cellulose and hemicellulose and can be regarded as a measure of the plant cell wall material. The method of determining NDF was originally devised for forages, but can also be used for starch-containing foods provided an amylase treatment is included in the procedure. The term non-structural carbohydrate (NSC) is sometimes used for the fraction obtained by subtracting the sum of the amount (g/kg) of crude protein, ether extract, ash and NDF from 1000. The method of assessing crude fiber involves refluxing and washing already defatted food sample with diluted acid and base to neutral point. There are many other techniques, the acid-detergent lignin determination involves the preparation of acid-detergent fibre as the preparatory step. The ADF is treated with 72% sulphuric acid, which dissolves cellulose. Ashing the residue determines crude lignin, including cutin. In human nutrition, the term dietary fibre is often used. This is defined as lignin alongside polysaccharides that cannot be digested by monogastric endogenous enzymes. Dietary fibre is difficult to assess in the

laboratory and the alternative term non-starch polysaccharides (NSP) has been adopted. The NSP in most food, along with lignin is considered to represent the major components of cell walls. The major constituents of NSP are rhamnose, arabinose, xylose, glucose, mannose and glucuronic and galacturonic acids. Cellulose is the major source of glucose, and hemicellulose provides xylose, mannans and galactose. The degradation of pectin releases arabinose, galactose and uronic acids (Samuel, 2016).

2.2.7 Energy

Energy is the amount of strength and vitality derived from food substances through the process of cellular respiration. It is measured in Joules (J). The energy food energy helps to sustain muscles and tissues. It can be derived from the proximate contents of energy rich foods especially wheat foods. The value of energy of a given food can be estimated base on their contents of crude proteins, fats and carbohydrate (Salau, 2012).

2.3 Mineral Analysis

Trace elements are both vital and non-vital in an organism. Essential trace elements are those elements that are required for the correct functioning and equilibrium of the organism. Non-essential trace elements are those that may be toxic to the organism. The trace element in plant gets by absorption from the soils or nutrient solutions through roots, foliar or translocation. Both essential and non-essential trace element becomes harmful when their levels increase. However, some of the elements are of healthful benefit to humans in low amount in crops. It is important for the identification of adequate, sub adequate and marginal intake levels for humans, so that diseases related to trace element deficiency can be prevented or minimized (Arewa, 2017).

2.3.1 Digestion method

The wet digestion method involves the chemical degradation of sample matrices in solution by addition of liquid reagents in order to solubilize it. The choice of acid has to be given consideration as certain samples or metal may not be oxidized and may be incompatible with certain metal elements. For example, sulfuric acid cannot be used to dissolve samples containing Barium, while hydrogen chloride cannot be used to dissolve samples containing Pb and Pb compounds. Oxidizing acids such as nitric acid, is more preferred due to its oxidizing ability. Thus, the metal analyte is converted to nitrate salts which are highly soluble and hence keeping the analyte in solution until they reach the plasma of the ICPMS or flame of the AAS.

2.3.2 Instrumental analysis

2.3.3 Atomic absorption spectroscopy

Atomic absorption spectrometry (AAS) is an analytical technique which determines the concentrations of elements. It was first employed as an analytical method, and the underlying principles were established in the second half of the 19th century by Robert Wilhelm Bunsen and Gustav Robert Kirchhoff, both professors at the University of Heidelberg, Germany (Zaidan *et al.*, 2018).

The method employs the principle which implies that the free atoms produced in the atomizer can absorb radiation of a particular wavelength. Thus, it quantifies the absorption of ground state atoms in the gaseous state, when they absorb UV (ultraviolet) or Visible light and make transition to the excited states. It is used to estimate the concentration of metal ions by analyzing the spectrum produced when a substance is vaporized and absorbs certain frequencies of light. It has high sensitivity; it can measure down to parts per billion of a gram (μgdm^{-3}) in a sample. Atomic absorption spectrometry has many applications in different areas of chemistry such as clinical analysis, environmental, pharmaceuticals,

industry and mining. The method uses absorption spectrometry in assessing the concentration of an analyte in a sample. It makes use of standard with known analyte content to establish a relationship between measured absorbance and concentration of the analyte and depends therefore on the Beer-Lambert Law (Gracia and Baez, 2012).

Before a sample is sent to the AAS instrumental analysis, digestion is first carried to obtain the metal analyte in a soluble form. This is then followed by the calibration of the instrument using a standard and blank to prepare a calibration curve. The soluble liquid obtained from the digestion process is introduced into the cuvette directly, where it is transformed into fine mist. It is then exposed to radiation originating usually from a source set at defined wavelengths to be absorbed by the analyte metal atoms in the sample. Thus, resulting in a light spectrum that has reduced intensity in one or more of its areas. The reduced intensity is characteristic of the analyte metal and hence, identifies and quantifies the metal. It is advantageous such that different radiation wavelengths are absorbed by different atoms. Its reliability is based on its ability to relate intensity of absorption to concentration with a simple line. It is cheap, fast and relatively easy to operate.

2.3.4 Flame emission spectroscopy

Flame emission is so named because it uses flame to provide the energy of excitation to atoms (Bejaoui *et al.*, 2014). It atomizes a sample solution into a flame, isolates the characteristics spectral emission of an element, and detects and measures this emission (Shaikh *et al.*, 2017).

This technique is sensitive, inexpensive and simple method for detecting common metals. In flame emission spectrometry, sample solutions are first nebulized (converted into a fine aerosol) and then introduced into the flame where they are desolvated, vaporized, and atomized, all in quick succession. This is then followed by the transition of atoms and

molecules to excited states by thermal collisions with the constituents of the partially burned or combusted flame gases. As they return to a lower or ground electronic state, radiation(s) characteristic of the excited atoms and molecules of the sample constituents are emitted. The emitted radiation passes through a monochromator that isolates the specific wavelength for the desired analysis. A photo detector measures the radiant power of these selected radiations, which is then amplified and directed to a read-out device or microcomputer system (Rogalski and Chirzanowski, 2017).

The use of combustion flames provides a means of vaporizing the analytes in solution into free atoms which are then excited by any of these two methods: absorption of further thermal energy from the flame or absorption of radiant energy from an external source of radiation. In the first method, known as flame emission spectroscopy (FES), the energy required to move the electrons of the free atoms from the ground state to excited states is supplied from the flame. The intensity of radiation emitted by these excited atoms returning to the ground state provides the basis for analytical determinations in FES. (L'vov, 2010).

2.5.3 Elements and importance

2.4.1 Magnesium

Magnesium aids in the conversion of food into energy and helps to make the parathyroid gland that produces hormones important for the bone's health. It is a cofactor for hundreds of enzymes, helps in protein synthesis, ion transport, cell signaling and structural functions. It also helps the body to absorb and decomposition of various other vitamins and minerals for example calcium and vitamin C. Bread, beans, nuts are good dietary source of magnesium. The current allowable magnesium intake in men is 400-420mg/day and 310-320mg/day for women. The adult human body is made up of about 25g of magnesium. 60%

of all the magnesium in the body is found in the bones, 27% in muscles, 6-7% in other cells and less than 1% outside the cells (Elzoghby *et al.*, 2014).

Magnesium is required for a series of step in the formation of deoxyribonucleic acid (DNA), ribonucleic acid (RNA), and proteins. Several enzymes involved in the formation of carbohydrates and lipids need magnesium for their activity. Glutathione, a very vital antioxidant requires magnesium for its synthesis. Magnesium plays a structural role in bone, cell membrane and chromosomes, it is required for the active transportation of calcium and potassium ions across the cell membrane. Through its ability ion transport, magnesium affects the conduction of nerve impulses, muscle contraction and normal heart rhythm (He *et al.*, 2016).

Magnesium deficiency results in gastrointestinal disorder such as prolonged diarrhea, crohn's disease, mal-absorption syndromes, celiac disease, surgical removal of a portion of the intestine, and intestinal inflammation due to radiation may all lead to magnesium depletion. Renal disorder for example, diabetes mellitus long-term use of certain diuretics may result in increased urinary loss of magnesium (He *et al.*, 2016). Several studies have found that elderly people have relatively low dietary intakes of magnesium (Stalmam *et al.*, 2011; Moshfegh *et al.*, 2019). As intestinal magnesium absorption tends to decrease with age, urinary magnesium excretion tends to increase with age; thus, suboptimal dietary magnesium intake may increase the risk of magnesium depletion in the elderly.

2.4.2 Iron

Iron is the central metal in the hemoglobin molecule for oxygen transport in the blood and is portion of myoglobin located in muscles.

Iron is an essential nutrient for transportation of oxygen and generation of cellular energy, and is a functional component/ cofactor in over 200 metalloenzymes. However, excess iron

is toxic because of Fenton chemistry-derived free radicals; consequently, iron homeostasis is highly regulated. (Wiley *et al.*, 2015).

Table 2.0. Recommended Daily Allowances (RDAs) for Iron.

Age group	Years of age	RDA (mg/day)
Children	2 – 11	13.7 - 15.1
Adolescent	12 – 19	16.3
Men	20 – older	19.3 - 20.5
Women	20 – older	17.0 - 18.9
Pregnant Women	18 – older	14.7

Source: Wiley *et al* (2015)

2.4.3 Phosphorous

Phosphorous is vital structural constituent of cell membranes and nucleic acids that involves series of biological processes, like bone mineralization, energy production, cell signaling through phosphorylation reactions, and regulation of a pH homeostasis (Elzoghby *et al.*, 2014). The RDA recommended dietary allowance of phosphorous is 70mg/day in adult. It is commonly found in most food source and also often component of most food additives. The availability of phosphorous is very high except for phylate phosphorous in plant sources such as grains, legumes and seeds with poor digestibility. Phosphorous is very

essential as it is the major structural components of bones in form of a calcium phosphate salt known as hydroxyapatite. It is one of the main structural components of cell membranes. All energy production and storage are dependent on phosphorylated compounds such as ATP and creatine phosphate. DNA and RNA are long chains phosphate molecules known for the storage and transmission of genetics information. Phosphate helps in the maintenance of the body acid-base balance by acting as a buffer in the body (Elzoghby *et al.*, 2014). Lack of proper intake of phosphorous rarely results in abnormally low serum phosphorous levels (hypophosphatemia) because renal reabsorption of phosphorous increases to compensate for decreased intake. The effects of moderate to severe hypophosphatemia may include loss of appetite, anemia, muscle weakness, bone pain, rickets, (in children), osteomalacia (in adults), increased susceptibility to infection, numbness and tingling of the extremities, difficulty walking and respiratory failure. Severe hypophosphatemia may occasionally be life threatening. Since phosphorous is so widespread in food, dietary phosphorous deficiency is usually seen only in cases where complete starvation is observed. Those prone to hypophosphatemia include alcoholics, diabetics recovering from an episode of diabetic ketoacidosis, patients with respiratory alkalosis and starving or anorexic patients on refeeding regimens that are high in calories but too low in phosphorus (Baxter *et al.*, 2010).

In food, phosphorus in plant seeds (beans, peas, cereals and nuts) is present in a storage form of phosphate called phytic acid or phytate. Only about 50% of the phosphorus from phytate is accessible to humans because we lack phytate enzymes that liberate phosphorus from phytate (Uribarri and Calvo, 2013). Yeasts contain phytates, so whole-grains incorporated into leavened breads have more bioavailability of phosphorus than whole-grains incorporated into breakfast cereals or flat breads (Hathcock *et al.*, 2017). Because

reducing dietary phosphorus absorption maybe helpful to individuals with impaired kidney function who are at risk of hyperphosphatemia (serum phosphorus at or above the high normal range), protein sources of phosphorus in grain-based vegetarian diets may be preferred over meat-based diets (Moe *et al.*, 2011).

2.4.4 Calcium

Calcium is one of the major components of bones and teeth and also plays a vital role as second messenger in cell signaling pathways. Circulating calcium concentrations are mainly controlled by the parathyroid hormone (PTH) and vitamin D at the expense of the skeleton when dietary calcium intakes are inadequate. The daily allowable intake of calcium is 1000-1,200 mg/day for adults. The world health organization advises that all pregnant women in areas of low calcium intake (that is low-income countries with intake around 300-600mg/day) maybe given supplemental calcium starting in the 20th week of pregnancy (Elzoghby *et al.*, 2014).

The skeleton is a reserve of calcium drawn upon to maintain normal serum calcium in case of inadequate dietary calcium. Thus, calcium sufficiency is required to maximize the attainment peak bones at old age, which leads osteoporosis, bone fragility and an increased risk of fractures. High concentration of calcium oxalate stones in the kidneys. Because dietary calcium intake has been inversely associated with stones occurrences, it is thought that adequate calcium consumption may reduce the absorption of dietary oxalate, thus reducing urinary oxalate and kidney stone. Adequate calcium intake is vital for maintaining healthy skeleton. Calcium is found in a variety of foods, including dairy products, tofu,

beans and vegetable of the kale family. The bioavailability of calcium among food is known and certain drugs are known to adversely affect calcium absorption (Sheng *et al.*, 2010).

Calcium is the most abundant mineral in the human body. About 99% of the calcium in the body is found in bones and teeth, while the other 1% is found in the blood and soft tissue. Calcium concentrations in the blood and fluid surrounding the cells (extracellular fluid) must be maintained within a narrow concentration range for normal physiological functioning. The physiological functions of calcium are so essential to survival that the body will stimulate bone desorption (demineralization) to maintain normal blood calcium concentrations when calcium intake is inadequate. Thus, adequate intake of calcium is a determinant factor in maintaining a healthy skeleton (Elzoghby *et al.*, 2014).

Calcium is one of the main structural elements in bones and teeth. It consists mainly of hydroxyapatite $[\text{Ca}_{10}(\text{PO})_6(\text{OH})_2]$ crystals which contain large amount of calcium, phosphorus and oxygen. Bone is a dynamic tissue that is remodeled throughout life. Bones forming cells called osteoblasts, begin the process of remodeling by dissolving or resorbing bone. Bone forming cells called osteoblasts then synthesize new bone to replace the bone that was resorbed. During normal growth, bone formation exceeds formation. Calcium is important as it aids the stabilization of a number of proteins, including enzymes optimizing their activities. The binding of calcium ions is required for the activation of the seven “vitamin K-dependent” clotting factors in the coagulation cascade. The term ‘coagulating cascade’ refers to a series of events each dependent on the other than stops bleeding through clot formation (Elzoghby *et al.*, 2014).

2.4.5 Sodium

Sodium in its salt form (sodium chloride) is essential for life. The right regulation of the body's sodium and chloride concentration is so important that multiple mechanisms work in concert to control them. Although scientists agree that a minimum amount of salt is needed for survival, the health repercussions taking excess of salt signifies an area of continued investigation among scientists, clinicians and public health experts (Diosi *et al.*, 2015). Sodium (Na^+) and chloride (Cl^-) are the principal ions in the extracellular fluid, which includes blood plasma. Hence, they play very vital roles in a number of life-sustaining processes (Harper, 2010). Sodium and chloride are electrolytes that help in the regulation of concentration and charge differences across cell membranes. Absorption of sodium in the small intestine plays an important role in the absorption of chloride, amino acid, glucose and water. Similar pathways are involved in the reabsorption of these nutrients after they have been filtered by the kidneys from the blood. Chloride, in the form of hydrochloric acid (HCl), is an important constituent of the gastric juice, aiding digestion and absorption of many nutrients in the body (Harper, 2010).

Because sodium is a primary determinant of extracellular fluid volume, including blood volume, a number of physiological pathways that regulate blood volume and blood pressure work by adjusting the body's sodium content. In the circulatory system, baroreceptors observe changes in blood pressure and send excitatory or inhibitor signals to the nerve system and/or endocrine gland to affect sodium regulation by the kidney. In general, sodium retention results in water retention and sodium loss results in water loss (Sheng *et al.*, 2010). Below are descriptions of two of the many systems that affect the blood volume and blood pressure through sodium regulation.

Sodium (and chloride) deficiency does not result from inadequate dietary intake, even in those on very low-salt diets (Baxter *et al.*, 2010). Hyponatremia, which is a serum sodium

concentration less than 136 mmol/litre, may occur from increased fluid retention (delusional hyponatremia) or increased sodium loss. Delusional hyponatremia may be due to inappropriate anti-diuretic hormone (ADH) secretion, which is associated with impairment affecting the central nervous system and with use of certain drugs. Excessive intake of water may also lead to delusional hyponatremia. Conditions that promote the loss of sodium and chloride include, severe or prolonged vomiting or diarrhea, excessive and persistent sweating, the use of some diuretics and some forms of kidney disease. Symptoms of hyponatremia include headache, nausea, vomiting, muscle cramps, fatigue, disorientation and fainting. Complication of severe and rapidly developing hyponatremia may include cerebral edema (swelling of the brain), seizure, coma and brain damage. Acute or severe hyponatremia may be critical without prompt and appropriate medical treatment (Adroque and Madias, 2010).

2.4.6 Potassium

Potassium is a vital mineral and electrolyte, tight regulation of potassium concentrations both inside and outside cells determines normal body function (Krejpcio *et al.*, 2011).

Potassium is a cation in the intracellular fluid, while sodium has a cation in the extracellular fluid. Potassium concentrations are about 30 times larger inside than outside cells, while sodium concentrations are more than ten times smaller inside than outside cells. The concentration differences between potassium and sodium across cell membrane create an electrochemical gradient known as membrane potential. This is maintained by ion pumps in the cell membrane, especially the sodium, potassium –ATPase pumps. These pumps use ATP (energy) to pump sodium out of the cell in exchange for potassium. Their activity has been estimated to account for 20% - 40% of the resting energy expenditure in a typical adult. The large proportion of energy dedicated to maintaining sodium/potassium

concentration gradients emphasizes the important of this function in sustaining life. Tight regulation of cell membrane potential is critical for nerve impulse transmission, muscular contraction and heart function (Sheng, 2010; Brody, 2014).

In 2004, the Food and Nutrition Board of the Institute of Medical established an adequate intake level for potassium based on intake levels that have been found to lower blood pressure, reduce salt sensitivity and minimize the risk of kidney stones (Hathcock *et al.*, 2017). The allowable daily intake for adult is 470mg/day for both male and female and 3000mg/day for children.

An abnormally low plasma potassium concentration is referred to as hypokalemia. Hypokalemia is the most commonly result from excessive loss of potassium, for example prolonged vomiting, the use of some diuretic in kidney disease cases, or metabolic disturbances. The symptoms of hypokalemia are related alteration in membrane potential and cellular metabolism. Such as fatigue, muscle weakness and cramps and intestinal paralysis that can lead to bloating, constipation and abdominal pain. Severe hypokalemia may result in muscular paralysis or abnormal heart rhythms (cardiac arrhythmias) that can fatal (Sheng *et al.*, 2010).

The diets of Western industrialized culture are quite different from those prehistoric cultures and the few remaining isolated primitive culture. Among other differences, the daily intake of sodium chloride (salt) in western industrialized cultures is about three times higher than the daily intake of potassium on molar basis, whereas salt intake in primitive cultures is about seven times lower than potassium intake (Robinson *et al.*, 2014). The relative deficiency of dietary potassium in the modern diet may play a role in the pathology of some chronic diseases.

2.4.7 Zinc

Zinc is a vital mineral element for human healthy body functioning, it is the second most abundant metal in the body after iron. It is highly essential for proper functioning of the body immune system, white blood cell formation, egg fertilization, cell division and a host of several enzymatic activities. It is required for proper action of insulin, smell and taste receptors. Zinc is needed for the development of infant, pregnancy and child growth. When foods containing required amount of zinc is consumed, it minimizes/prevents the possibility of diseases like the common cold (Wiley *et al.*, 2015).

Zinc is also important in the development of and function of male sex organs. Males with zinc deficiency have portrayed less developed testes and reduced sperm count. Thus, zinc supplements may be used as potential treatment for erectile dysfunction as it aids the synthesis of key sex hormones such as testosterone and prolactin. The recommended daily allowance (RDA) established for boys and men age 14 and older 11mg/day; women age 19 and older, 8mg/day; pregnant women age 19 and older, 11mg/day; lactating women age 14 to 18, 14mg/day; lactating women age 19 and older, 132mg/day (Soetan *et al.*, 2010).

2.5 Physical Quality Measurement

2.5.1 Farinography

This is the measurement of the properties of flour using an apparatus (farinograph) that aids continuous assessment of torque in the shearing of polymer with a range of shear rates. Farinograph was designed by Carl William Brabender and produced by Brabender industries founded in 1923. The farinograph measures the shear and viscosity of a fluid, and also records the torque developed by the action of the mixer blades on the dough during mixing. It aids in the prediction of water absorption of flours, the relative mixing time, the stability of over mixing and rheological properties of the dough during mixing (Seribu and Arghire, 2017).

During dough mixing, the curves plotted mainly consist of an increase in resistance-to-extension (increased curve height) to an identifiable peak followed by a decline that reflects a decrease in resistance-to-extension. Thus, the curves for weak and strong flours show significant differences.

Mixing is performed by two sigma-shaped blades rotating at a differential speed of 3.2. The temperature condition of the whole process is maintained at 30°C by a thermostatically controlled circulation water bath that pumps water through a cavity in the mixing bowl. The mixing bowl can be designed for 50g or 300g flour sample. A dynamometer drives the mixing blades and the torque developed is transmitted by a lever system to a scale and recording mechanism (Huang *et al.*, 2020). The following properties are observed during farinograph analysis;

2.5.2 Arrival time

This is the time to the nearest half minute from the first addition of water until the top of the curve first intersects the 500 B.U. consistency line (hydration time).

2.5.3 Departure time

This is the time to the nearest half minute from the first addition of water until the point where the top of the curve leaves the 500 B.U. line. Thus, departure time determine the stronger flour.

2.5.4 Mixing tolerance index

This is the difference in Brabender unit from the top of the curve at the peak to the top of the curve measured five minutes after the peak is reached. It indicates the mixing tolerance of flour, an MTI value of 30 B.U. or less indicates excellent for hard wheat flours. A flour with MTI value >50 B.U. indicates less tolerance and often shows difficulty in mechanical handling and make up of dough.

2.5.5 Stability time

This is the difference in time to the nearest half minute between the arrival time and the departure time. It indicates the suitability of flour for hearth or varieties of bread production and often requires longer mixing time at high stability.

2.5.6 Water absorption capacity

This is the amount of water required to develop standard dough consistency of 500 B.U. at the peak of the curve which ranges between 55 and 62% (Wilson *et al.*, 2018).

2.6 Sedimentation Analysis

This is an analysis carried out to assess the particle size distribution of samples. According to Zeleny, it reveals the degree of particle size distribution when suspended in lactic acid solution during a standard time interval. Hence, the quality of wheat flours can be assessed as it reveals the possibility of adulteration.

Sedimentation involves the hydration of a small flour sample in lactic acid solution having the detergent either sodium dodecyl sulphate (SDS sedimentation) or isopropyl alcohol (Zeleny sedimentation). Hydrated flour particles sink in the form of sediment in the presence of lactic acid solution. The sedimentation volume (SV), shows the strength of gluten as high SV indicates strong gluten while low SV indicates weaker gluten. According to Karaduman *et al* (2019), lactic acid ruptures the cell-wall of the endosperm leading to hydration of the particles of the endosperm, thus causing the formation of proteinaceous fibrillation molecules binding the flour particles together. The stability of the fibril structure is enhanced by the disulphide linkage existing between the gluten molecules (Carter *et al.*, 2016).

2.7 Chemometrics

Chemometrics encompasses the use of mathematical and statistical means to improve the understanding and appreciation of chemical data by correlating the quality parameters or physical properties to analytical instrument data. Patterns in the data are first modeled; these models are then successively applied to future data in order to predict the same quality parameters. The result of the chemometrics approach has a renowned ability in assessing product quality. It enhances more efficient laboratory practices by optimizing experimental procedures and equipment calibration and encourages automated quality control systems. The only requirements are a suitable instrument and the software to interpret the patterns in the data. (Semih and Vasfiyen, 2015).

Chemometrics as a science gives spectroscopist numerous efficient opportunities to solve the calibration challenges for analysis of spectral data. It can be used to improve methods development by employing routine use of statistical models for data analysis. Spectroscopists use software packages such as The Unscrambler for spectroscopic data analysis, classification, modeling and prediction to meet process monitoring and quality assurance needs.

2.7.1 Principal component analysis

This is the mathematical manipulation of the row space of the food sample matrix against the mineral content variables. It simplifies the multivariate view of drawers to enhance human structural identification and pattern recognition. It encompasses several other plots Such as the score plots, loading plots, Eigen value plots, Hotelling T^2 and Q-residual plots and nipples. Each has its own unique characteristics with which they contribute to the latent understanding of the chemical data obtained (Chudzinska and Baralkiewicz, 2010).

2.7.2 Eigen value plots

The Eigen value plots gives clue to the selection of principal components that will capture the major variables responsible for the sample attribute depending on the magnitude of the factors in the data set that has to be accounted for.

2.7.3 Score plots

The score plot provides information about patterns existing amidst samples. It reduces the difficulty in visualizing the similarities, patterns, groupings and differences existing among the studied foods.

2.7.4 Loading plots

The loading plots enhance the understanding of the principal factors responsible for variation among samples and also aids proper interpretation of data based on their properties. Loading plots reveals the basis of groupings, gives information regarding correlating elements which in turn helps one to visualize and characterize the general food qualities.

2.7.5 Hotelling T^2 and Q-residual plot

The Hotelling T^2 and Q-residual plot is employed to explain exceptional behavior. It is a Statistical concept used in capturing the significance of multivariate distances. It is a diagnostic tool use for detecting outlier sample. Thus, it identifies the score or samples that are usually far from or less correlated with other samples. It summarizes the extent to which principal component analysis (PCA) model describe the sample scores by indicating the distance of each sample from the center of the model (at score= 0).

2.7.6 Biplot sketch

The biplot sketch helps in bringing out simultaneous information about samples (in score plot) at the same time with variables (in loading plot). It reveals information regarding the variable that correlates or is an important factor of the analytes. It permits the subgroupings of the samples into various categories. Thus, enhancing proper identification.

2.8 Cluster Analysis

Cluster analysis is a form of data reduction technique. Data reduction analyses, which include discriminant analysis and factor analysis, essentially reduce data just as the name implies. They do not analyze group differences on the basis of independent and dependent variables. For example, factor analysis decreases the number of variables or factors within a model, while discriminant analysis classifies new cases into the groups that have been previously identified based on some specific criteria (Daniel *et al.*, 2011). Cluster analysis is unique among these other techniques because its aim is to reduce the number of observations or cases by classifying them into homogeneous clusters, identifying groups without previously knowing group membership or the number of possible groups. Cluster analysis also allows for many options regarding the algorithm for combining groups, with each choice resulting in a unique grouping structure. Therefore, cluster analysis is a convenient statistical tool used in exploring underlying structures in various kinds of datasets. Cluster analysis was first employed within the fields of biology and ecology (Pelninger *et al.*, 2019). Although this technique has been used in the social sciences, it has not gained as much popularity as in the physical and natural sciences.

2.8.1 Hierarchical cluster analysis

Hierarchical clustering combines cases or observations into homogeneous clusters by merging them together one at a time in a series of sequential steps (Yim and Ramdeen, 2015). The other variant, the nonhierarchical techniques first establish an initial set of

cluster means and then assign each case to the closest cluster mean (Morissette and Chartier, 2013). At each step in the hierarchical process, a new cluster is formed or one case joins a previously grouped cluster. All the steps are irreversible meaning that cases cannot be reassigned to a different cluster afterwards. This makes the initial clustering steps extremely influential due to the fact that the first clusters generated will subsequently be compared to all of the other cases. The alternate method of non-hierarchical clustering requires a researcher to establish a priori the number of clusters in the final solution. If there is uncertainty regarding the total number of clusters in the dataset, the analysis must be re-run for each probable solution.

Hierarchical cluster analysis can be agglomerative or divisive. Agglomerative hierarchical clustering separates each of the cases into its own individual cluster in the first step so that the initial number of clusters equals the total number of cases (Margaritis *et al.*, 2020). At successive steps, similar cases or clusters are merged together until every case has been grouped into a single cluster.

Divisive hierarchical clustering works in the reverse fashion with every case starting in a single large cluster before gradually being separated into groups of clusters until each case is in an individual cluster. This divisive clustering technique is rarely used due to its heavy computational load.

2.8 Matlab

Matrix laboratory is a multi-paradigm numerical computing environment and proprietary programming language developed by MathWorks. MATLAB allows matrix manipulations, plotting of functions and data, implementation of algorithms, creation of user interfaces, and interfacing with programs written in other languages, including C, C++, C#, Java, Fortran and Python.

Although MATLAB is intended primarily for numerical computing, an optional toolbox uses the MuPADsymbolic engine, allowing access to symbolic computing abilities. An additional package, Simulink, adds graphical multi-domain simulation and model-based design for dynamic and embedded systems. As of 2018, MATLAB has more than 3 million users worldwide (TMW, 2018). MATLAB users from various backgrounds of engineering, science, and economics.

CHAPTER THREE

3.0

MATERIALS AND METHODS

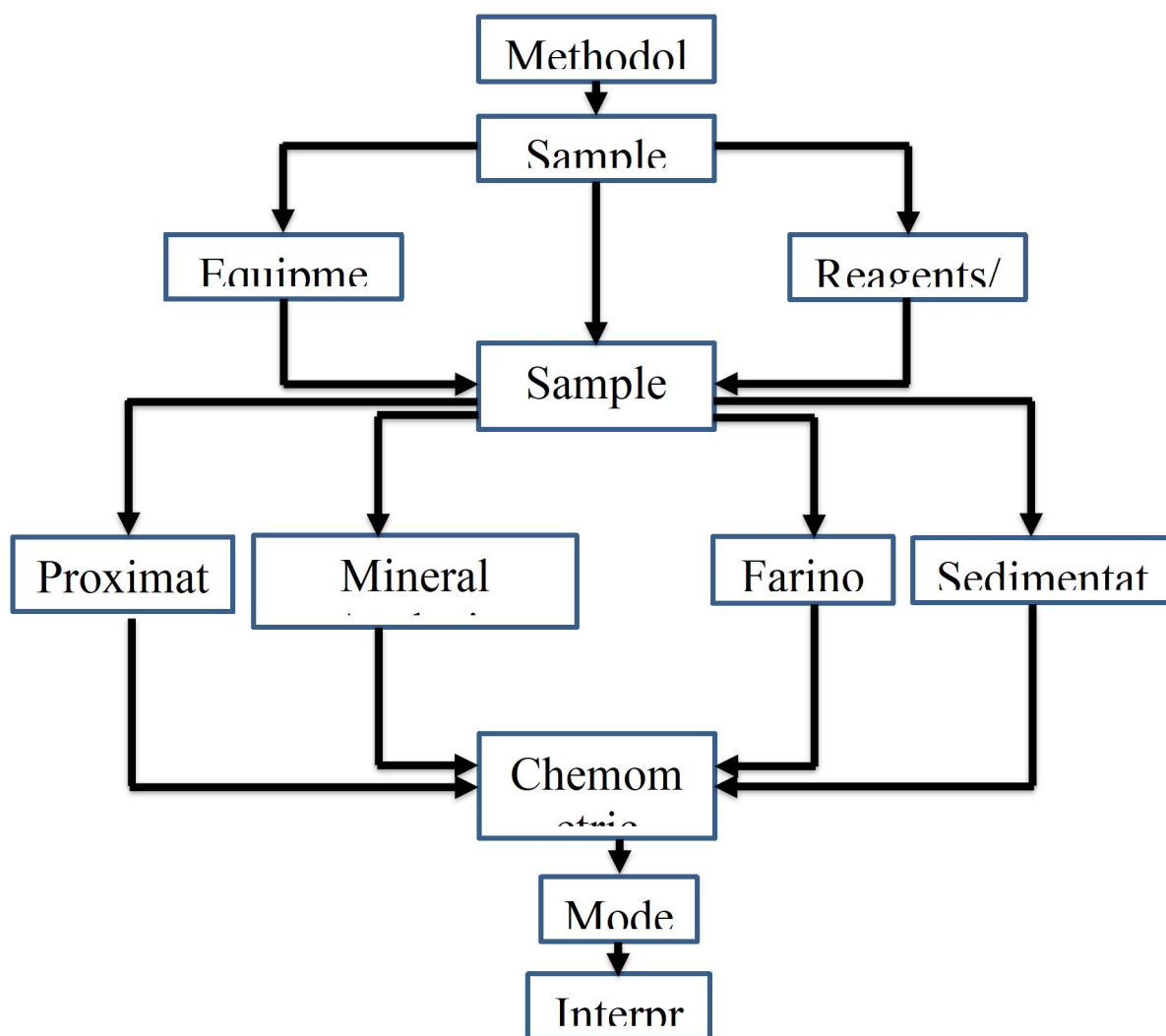


Figure 3.1 Methodology framework.

3.1 Materials Used

Table 3.1: Apparatus and equipment used

Name	Model	Place of Manufacture
------	-------	----------------------

Beakers	-	-
Conical flasks	-	-
Funnels	-	-
Filter paper	-	-
Retort stand		
Ceramic mortar and pestle	-	-
Weighing balance	AE160	England (J.M. Bird)
Hotplate	-	U.S.A (Narcis)
Flame photometer	-	England (EWGISE)
Kjedahl apparatus	-	U.S.A (LOOMIS CO)
Farinograph apparatus	-	Duisburg, Germany
Soxhleht apparatus	-	England (EWGISE)
Atomic absorption spectrometer	(AA5000)	U.K (Spectrum)
		-
Colorimeter		U.K (Spectrum)
Centrifuge		-
Software		Microsoft Excel 2013
		Inc, MATLAB
		version7.6.0 and PLS
		Tool Box

Table 3.2 Chemicals used in the study

Reagents	Purity	Manufacturer
Sulphuric acid	99.5%	British Drug House
Hydrogen peroxide	98%	Sigma Aldrich
Deionized water	99%	British Drug House
Sodium tungstate	94%	TNJ Chemical Industry
Fehlings solution	98%	British Drug House
Copper sulphate	90%	May and Baker, England
Lactic acid	99%	British Drug House
Hydrochloric acid	98%	British Drug House
Trichloroacetic acid	99.5%	TNJ Chemical Industry
Molybdate reagent	99%	Sigma Aldrich

3.2 Sampling and Sample Preparation

A total number of 14 wheat product samples were obtained from 3 major markets in Minna (Kure market, Kasuwan Gwarri and Tunga Markets) Niger State. The samples collected were transported to the laboratory of Chemistry Department, Federal University of Technology Minna Niger State for further analysis.

Table 3.3 Sample description

Sample	Sample name	Description
1	WHI(Kure Market)	Raw wheat grain of common wheat species.
2	GP1 flour(golden penny)	General purpose wheat flour produced by Golden penny.
3	GP2 flour (honeywell)	General purpose wheat flour produced by Honeywell.
4	GP3 flour (mix n bake)	General purpose wheat flour produced by mix n bake.
5	WH4 (Kure Market)	Raw wheat grain of common wheat species.
6	GPF flour (Dangote).	General purpose wheat flour produced by Dangote.
7	SP1 (dangote)	Long thin string-like solid pasta made from durum wheat Produced by Dangote.
8	SP2 (Golden penny)	Long thin string-like solid pasta made from durum wheat produced by Golden penny.
9	SP3 (Bua pasta)	Thin long string-like solid pasta produced by Bua
10	SP4 (power pasta)	Thin long sting-like solid pasta produced by Bua past
11	WH2 (Kasuwan Gwarri)	Raw wheat grain of common wheat species
12	WH3(Tunga market) (Honeywell)	Raw wheat grain of common wheat species
13	SM1 (golden penny)	Coarse, purified wheat middlings of durum wheat Produced by Golden penny.
14	SM2 (Honeywell)	Coarse, purified wheat middlings of durum wheat Produced by Honeywell.

3.3 Methods

3.3.1 Wet digestion

The wet digestion procedure described by Jones and Case (1990) was adopted for the digestion of the samples. 1.0 g of sample was placed in a 250cm³ digestion tube and 3.5cm³ of concentrated H₂SO₄ was added. The mixture was allowed to stand for 30 min at room temperature. About 3.5cm³ of 30% H₂O₂ was added to the digestion tube and the sample was then heated at 250°C for 30 min. Thereafter, the digestion tube was removed from the digestion block and cooled down. 1cm³ of 30% H₂O₂ was added until the digest was clear upon cooling. When the solution was clear following cooling, it was filtered through Whatman No. 42 filter paper and transferred quantitatively to a 100 cm³ volumetric flask by adding deionized water.

3.4 Instrumental Analysis

3.4.1 Flame emission spectroscopy

The flame emission spectrophotometer was used to determine the concentration of Na, Mg, K and Ca elements at their corresponding wavelengths 589.0 nm, 285.2 nm, 765.5 nm, and 422.6 nm respectively. The sample solutions were sprayed or aspirated into the flame as fine mist or aerosol. The sample is vaporized in the flame and atomized by a combination of heat and a reducing gas. The atoms were excited into higher energy states by the heat which leads to emission of photons as they return to ground states. These photons were then measured by the detector.

3.4.2. Atomic absorption spectroscopy (AAS)

AAS was used to determine the absorption of Fe (248.3 nm) and Zn (213.9 nm) elements in each sample at their corresponding wavelengths.

3.4.3 Preparation of standard

This involves the preparation of range of standard solution of each analyte element by dilution of the stock solution. The various concentrations were made in the linear range of the instrument and appropriate for the amounts of elements likely to be present in the food extract.

The sodium and potassium standard solutions were prepared as described

Preparation of working standards

$$\begin{aligned} 1000 \text{ mg/dm}^3 &= \frac{\text{KCl}}{\text{K}} \text{ or } \frac{\text{NaCl}}{\text{Na}} & 3.1 \\ &= \frac{74.5}{39} \text{ or } \frac{58.5}{23} \\ &= 1.910 \text{ g/dm}^3(\text{K}) \text{ or } 2.543 \text{ g/dm}^3(\text{Na}) \end{aligned}$$

The calculated masses were dissolved in separate volumetric flasks and made to the mark to give 1000 g/dm³ stock solutions. The solutions were further diluted to 100 mg/dm³ by pipetting 10 cm³ of each of the stock solution and making it up to 100 cm³ with distilled water. Working standard of 0, 2, 4, 6, 8 and 10 mg/dm³ were prepared from 100 mg/dm³ (Ndamitso *et al.*, 2010).

Dilution method

$$C_1 V_1 = C_2 V_2 \quad 3.2$$

C_1 = initial concentration

C_2 = final concentration

V_1 = initial volume

V_2 = final volume

To prepare 100 dm³ from 1000 dm³ in 250 cm³

$$1000 \times V_1 = \frac{100 \times 250}{1000}$$

$$V_1 = 25 \text{ cm}^3$$

The standard solutions for Ca, Zn, Fe and Mg were purchased as industrial standards.

3.4.4 Calibration

The atomic absorption spectrophotometer was set according to the manufacturer instruction and the wavelength was set to that of the element to be analyzed. The meter was set to zero using blank solution and the absorbance of each standard solution was measured. Similarly, the absorbance of the digest was measured and if found to be too high, a known volume of the digest was diluted with water and the measurement was repeated while putting their dilution factor into consideration.

3.4.5 Colorimetric determination of phosphorus

Reagents of ammonium vanadate and ammonium molybdate were prepared by dissolving 0.625g of ammonium vanadate in 125 cm³ of distilled water followed by the addition of 5 cm³ concentrated trioxonitrate (V) acid and then diluted in 250cm³ volumetric flask with distilled water. Ammonium molybdate reagent was prepared by dissolving 12.5g of ammonium molybdate crystals in warm distilled water, the solution was then diluted to reach 250 cm³ volumetric flask. 20cm³ of each digest was transferred to a 250 cm³ volumetric flask followed by the addition of 100cm³ of distilled water, 40cm³ of each of the reagents prepared and then prepared made up to 250cm³ mark of the flask. The standard concentration was made up of potassium di-hydrogen phosphate containing dilute trioxonitrate (V) acid and same amounts of the two reagents. Absorbance was carried out

first, with blank and then standards at 465nm wavelength using 1cm cell (Salau and Hasan, 2014).

3.5 Proximate composition

All the parameters collected under this section were determined using the AOAC (2006) methods. The parameters analyzed were; moisture content, ash, crude fibre, crude protein, energy content, carbohydrate and crude fat (Arewa, 2017).

3.5.1 Total moisture content

The moisture content of the samples was determined in an oven by drying method (at 105 °C) as described in AOAC (2006) Method No. 44-15A. The moisture contents of the samples were determined by weighing 2g of sample into a known weight of filter paper and drying it in an air forced draft oven at a temperature of 105 ± 5 °C till the constant weight of dry matter was obtained. The moisture content was determined as follows-

$$\text{Moisture content (\%)} = \frac{w_1 - w_2}{w_1} \times 100 \quad (3.3) \quad \text{nt \%} = w_1 -$$

$$w_2 / w_1 \times 100 \quad (3.3)$$

Where;

W1 = weight of original sample before drying

W2 = weight of dried sample after drying

3.5.2 Determination of ash content

Ash is the inorganic residue remaining after the material had undergone complete combustion at a temperature of 550 °C in a muffle furnace. It is the aggregate of all non-volatile inorganic elements. About 8 g of finely ground dried sample was weighed into a porcelain crucible and incinerated at 550 °C for 6 hours in an ashing muffle furnace until

ash was obtained. The ash was cooled in a desiccator and reweighed. The % ash content in the sample was calculated as follows:

$$\text{Ash content (\%)} = \frac{W_1 - W_2}{W_1} \times 100 \quad (3.4)$$

Where;

W1 = weight of original sample before ashing

W2 = weight of sample after ashing

3.5.3 Determination of crude protein

Protein content was determined using the macro-Kjeldahl method as described by AOAC (2006). 0.5g of sample (dried) was weighted into 500 cm³ Kjeldahl flask; 20 cm³ of concentrated tetraoxosulphate (VI) acid will be added gently to each of the samples in the flask and it was heated on a heating block starting with a low heat about 200°C for 30 minutes and it was swirl by shaking the Kjeldahl flask occasionally to mixed and dissolve well. The temperature was increased to about 335 °C and was heated for about 5-6 hours to obtain a clear digest (complete digestion). Then it was switched off and allowed to cool, and diluted with 100cm³ of distilled water. 10 cm³ of boric acid was added into 100cm³ collection flask with 2 drops of mixed indicator and placed under the collection spigot of the distillation apparatus. 10cm³ of the digest was pipetted into the micro-distillation apparatus and 10ml of sodium hydroxide was added gently into the micro- distillation apparatus to react with the sample. The solution was allowed to distil for about 5 minutes or when the volume of ammonia collected in boric acid in the receiver flask was 50cm³ and the purple had turned green in color. The distillate was titrated against 0.1mol/dm³ hydrochloric acid to give a reddish color. A blank titration was carried out and percentage protein was calculated.

$$\% \text{crude protein} = \%N \times 6.25 = M \times \frac{V}{W} \times 14gN \times 100 \quad (3.5) = \%N \times$$

$$6.25 = M \times V/W \times 14gN \times 100 \quad (3.5)$$

Where;

M = molarity of HCl (mol/dm³)

V = corrected volume of acid (mol std. acid for sample – mol std. acid for blank)

W = weight of sample digested

14 = atomic weight of nitrogen

3.5.4 Determination of crude fibre

Ten grams (10g) of the wheat sample was weighed into 500 cm³ beaker and 200 cm³ of boiling 0.255 mol/dm³ tetraoxosulphate (VI) acid (1.25 percent w/v) was added. The mixture was boiled for 30 min keeping the volume constant by the addition of hot water at frequent intervals (a glass rod stirrer was placed in the beaker to enable smooth boiling). At the end of this period, the mixture was filtered through a muslin cloth and the residue washed with hot water till free from acid. The material was then transferred to the same beaker and 200 cm³ of boiling 0.313 mol/dm³ (1.25 percent w/v) NaOH was added. After boiling for 30 minutes, the mixture was filtered to a crucible, dried overnight at 80-100°C and weighed. The crucible was placed in a muffle furnace at 550 for 3 hours. It was then cooled in a desiccator and weighed again. The difference in residue weights and ash was taken as the weight of crude fiber (Sumczynski *et al.*, 2015).

3.5.5 Carbohydrate content

The total percentage carbohydrate content in the sample was determined by adding the total values of crude protein, lipid, crude fibre, moisture and ash constituents of the sample and

subtracting it from 100. The value obtained was taken as the percentage carbohydrate constituent of the sample. Thus:

$$\% \text{Carbohydrate} = 100 - (\% \text{ moisture} + \% \text{ crude fibre} + \% \text{ protein} + \% \text{ lipid} + \% \text{ ash}) \quad (3.6)$$

3.5.6 Determination of energy

The energy values of the samples were determined by multiplying the protein content by 4, carbohydrate content by 4 and fat content by 9.

$$\text{Energy Value} = (\text{Crude protein} \times 4) + (\text{Total carbohydrate} \times 4) + (\text{Crude fat} \times 9) \quad (3.7)$$

3.5.7 Determination of crude fat

The crude lipid in the powdered sample was determined using Soxhlet extraction for 24 hours. Approximately, 2.0 g of samples were weighed accurately into labeled thimbles in each case. The dried boiling flasks (250 cm³) were weighed correspondingly and filled with about 150 cm³ of petroleum ether (boiling point 40 -60 °C). The extraction thimbles were plugged tightly with cotton wool. After that, the Soxhlet apparatus was assembled and allowed to reflux for 24 hours. The thimble was removed with care and petroleum ether collected from the top container and drained into another container for re-use. After that, the boiling flask was heated in a hot air oven until it was almost free of petroleum ether. After drying, was cooled in a desiccator and weighed. The % fat in the sample was calculated using the formula:

$$\text{Fat (\%)} = \frac{w_1}{w_2} \times 100 \quad (3.8)$$

Where;

W1 = weight of fat

W2 = weight of original sample

3.6 Farinograph Analysis

A flour sample of 300 grams on a 14% moisture basis was weighed and placed into the corresponding farinograph mixing bowl. Water from a burette was added to the flour and mixed to form a dough. As the dough was mixed, the farinograph recorded a curve on the graph paper. The curve was centered on the 500-Brabender Unit (BU) line ± 20 BU by adding the appropriate amount of water and was run until the curve left the 500-BU line.

3.7 Sodium dodecylsalicylate (SDS)-Sedimentation Volume Test

3.7.1 Sedimentation reagents

1. Lactic acid solution: 3 cm³ of 88% lactic acid was diluted (1: 8 v/v) to 27 cm³ with distilled water.
2. SDS solution (2%): For this, 20g SDS (Sodium dodecyl sulphate) was dissolved in distilled water in 1 dm³ volumetric flask and made up to the mark.
3. The SDS-lactic acid reagent is prepared by dissolving SDS (20g) in distilled water (1litre) and then adding a stock diluted lactic acid solution (20cm³; 1-part lactic acid plus 8 parts distilled water by volume).

3.7.2 Sedimentation procedure

The sodium dodecyl sulphate (SDS) sedimentation volume of flour samples were estimated according to Ali *et al.* (2015). Five grams (5g) of flour (14% moisture basis) was added to

water (50 cm³) in a cylinder, a stop clock was started and the material dispersed by rapid shaking for 15 s. The contents were re-shaken for 15 seconds at 2- and 4-minutes interval immediately, following the last shake, SDS-lactic acid reagent (50 cm³) was added, and mixed by inverting the cylinder four times before re-starting the clock from zero time. Inversion was repeated four times before finally starting the clock once again from zero time. The contents of the cylinder were allowed to settle for 40minutes before reading the sedimentation volume.

3.8 Statistical Analysis

Descriptive analysis tool was used to generate output which includes; Mean and standard deviation of the total mineral contents and proximate contents. The correlation analysis was also carried out.

3.9 Chemometric Analysis

The data obtained from the proximate composition, mineral content and farinograph analysis were properly and sequentially imputed in microsoft excel format for chemometric analysis using the MATLAB Window and PLS tool box. The imputed data were used to carry out the principal component analysis (PCA) and hierarchical cluster analysis (HCA).

Data capturing: This was carried out by first preparing the chemical data obtained into a Microsoft Excel spread sheet.

Data processing: The desired PLS tool was chosen and the already captured excel data was imported, preprocessed and then auto-scaled.

Building of model: This involves choosing the appropriate principal component that will suit or enhance the latent plotting of the data set employed. The principal component 2 was adopted as it gives clearer information and plots of chemical data.

Validation of model: Having built a suitable model, the required analysis such as PCA or HCA was carried out. Plots such as Eigen, Score, Hotelling T^2 , biplot (PCA) or a dendrogram as observed in HCA were obtained.

3.9.1 Softwares used for the chemometric analysis

The three-software employed prior to the chemometric analysis are microsoft excel 2013Inc, MATLAB (version 7.6.0) and PLS tool box. This software enhanced efficient capture and interpretation of the chemical data obtained from proximate composition, mineral content and farinograph analysis to provide latent information in regard to their nutritional properties.

3.9.2 Principal component analysis (PCA) and hierarchical cluster analysis (HCA)

The PCA and HCA tool in the PLS Toolbox was activated by typing the following command in the MATLAB window:

Data matrix was Workspace Browser Window which directly show the analysis tool. Choose the clustering option and click on HCA loaded using the File/Imported data/Excel File to display the file. Type the command `>>browse` to open PLS

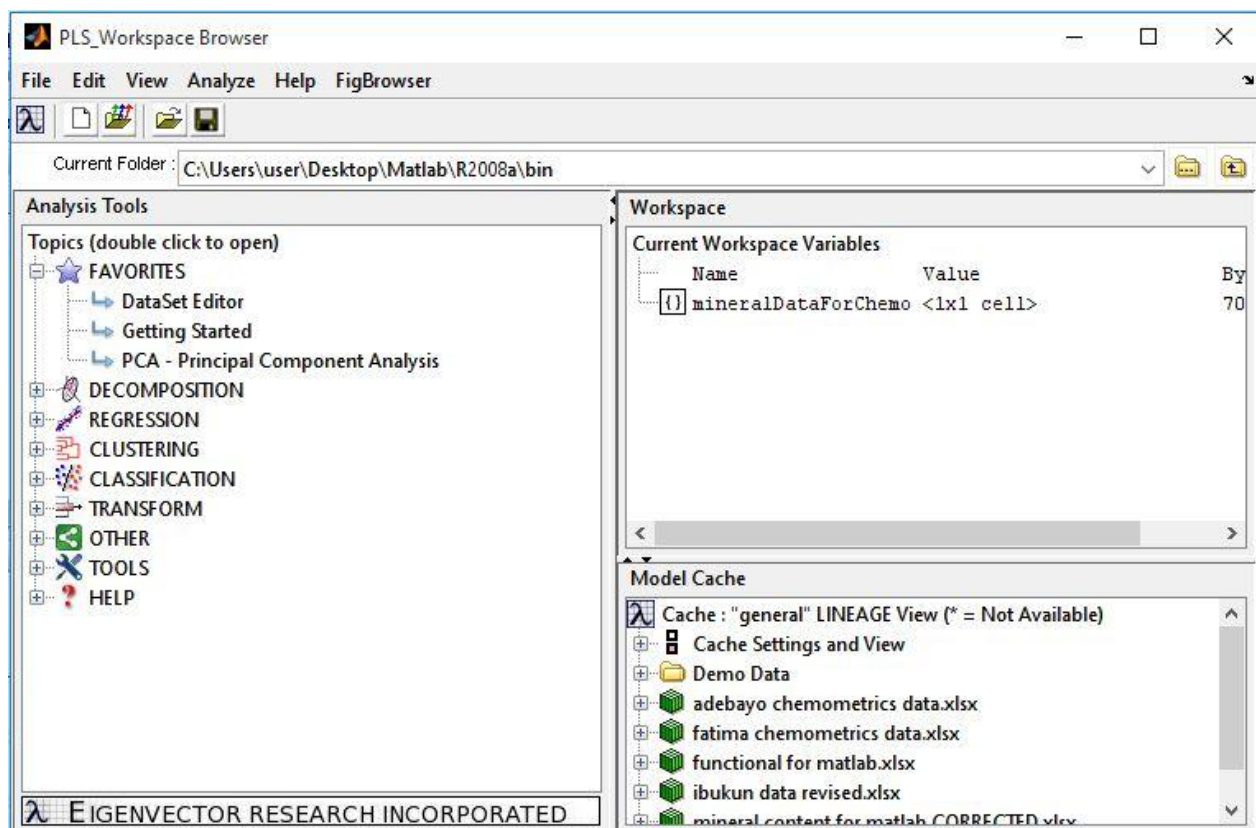


Fig. 3.2 PLS Workspace window

The modeling was performed by clicking on the model button. This is followed by the preprocessing mode then clicking on the preprocessing button. This technique affords a group of orthogonal axes that represents the direction of greatest variance in the data. The PCs 2 was chosen in this case and activated by clicking the apply button.

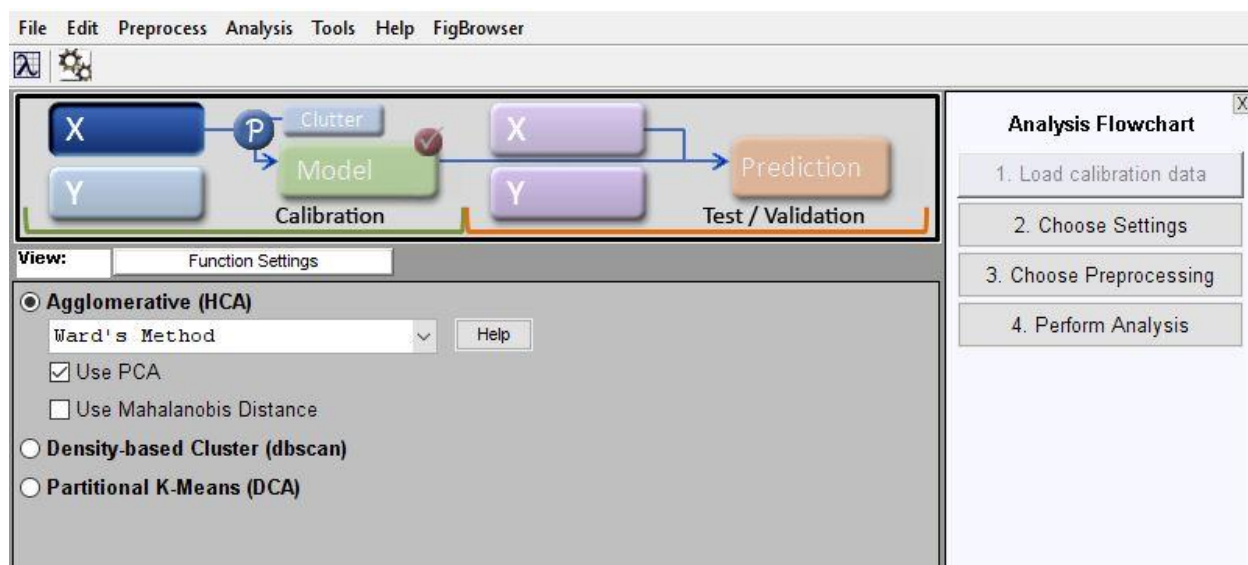


Fig. 3.3 Hierarchical cluster window

The Hierarchical Cluster Analysis enable the grouping of the samples based on similarity and dissimilarity. It also gives information on the relationship that exist between the samples.

CHAPTER FOUR

4.0

RESULTS AND DISCUSSION

4.1 Proximate Composition

Table 4.1 the proximate compositions of the unprocessed and processed wheat product samples. The ranges of the proximate compositions were as follows; crude fat 1.99-6.92%, moisture content 8.19-13.74%, crude protein 12.25-16.61%, crude fibre 0.69-1.16%, ash content 0.28-1.80%, carbohydrate 64.90-70.09% and energy 329.32-383.26kCal.

The crude fat contents ranged from 1.99-6.92%. The unprocessed wheat samples have the highest crude fat content in the following range (WH1 6.92- WH2 5.59%) followed by the processed semo samples (SM2 5.16 - SM1 4.64%) and SP1 (spaghetti sample) had the lowest (1.99%). The values for crude fat observed in this study were found to be in close agreement to that reported by Balarabe *et al.* (2017), which ranged from 1.49 to 6.5% in a physicochemical and sensory evaluation of wheat breads produced from various indigenous yeast isolates. The higher percentage of fat content in the unprocessed wheat samples indicates appreciable amounts of calories compared to the processed sample. The moisture (%) content ranged from 8.83-13.74%. The processed wheat samples were observed to have higher moisture content compared to the unprocessed wheat samples, GP4 has the highest moisture content (13.74%), followed by SM1 (13.02%) and WH1 was observed to have the lowest of the moisture content in all samples (8.19%). These values were found to be within the range reported by Balarabe *et al.* (2017) (0.24 - 24.1%).

Table 4.1: Proximate Composition of Wheat Samples

S/N	Sample	Crude Fat%	Moisture%	Crude Protein%	Crude Fiber %	Ash%	Carbohydrate%	Energy kCal
1	GP1	4.52±0.01	10.96±0.03	16.61±0.01	0.89±0.02	1.07±0.02	64.90±0.01	367.66
2	GP2	3.59±0.11	11.83±0.03	15.91±0.04	0.85±0.03	0.71±0.01	68.80±0.11	355.45
3	GP3	3.41±0.02	12.47±0.02	15.37±0.11	0.73±0.02	1.08±0.10	66.72±0.03	343.79
4	GP4	2.87±0.08	13.74±0.04	14.91±0.04	0.82±0.01	0.92±0.01	66.83±0.04	343.77
5	SP1	1.99±0.01	10.58±0.03	12.35±0.02	0.79±0.04	1.28±0.02	66.80±0.03	329.32
6	SP2	5.80±0.03	8.83±0.11	14.80±0.00	0.81±0.02	1.21±0.02	69.03±0.05	377.26
7	SP3	4.62±0.10	10.65±0.08	15.71±0.03	0.88±0.02	0.49±0.01	67.87±0.02	364.93
8	SP4	4.61±0.04	9.80±0.01	15.75±0.03	0.75±0.20	0.82±0.03	68.27±0.01	366.59
9	WH1	6.92±0.41	8.19±0.08	15.70±0.05	1.01±0.04	1.80±0.01	67.84±0.03	383.95
10	WH2	5.59±0.02	10.26±0.03	14.80±0.06	1.05±0.02	1.76±0.01	67.86±0.01	370.58
11	WH3	6.19±0.03	11.00±0.05	14.91±0.05	1.13±0.02	1.44±0.01	66.39±0.09	369.71
12	WH4	6.16±0.02	7.17±0.01	15.75±0.01	1.16±0.00	1.59±0.05	70.09±0.01	373.38
13	SM1	4.64±0.04	13.02±0.03	12.25±0.05	0.72±0.04	0.28±0.02	66.81±0.03	351.54
14	SM2	5.16±0.03	11.05±0.01	15.78±0.04	0.69±0.04	0.43±0.02	66.91±0.03	365.53

Key: The values are triplicate determinations \pm standard deviations (SD) for all the samples. GP1-GP4 = processed general purpose flour, SP1-SP4 = Spaghetti samples, WH1-WH 4= unprocessed wheat samples and SM 1-SM2= semo processed samples.

These values were observed to be higher than the result reported by Samuel (2016), of 8.64% from the assessment of the quality and sensory properties of wheat and sorghum flour cookies. The higher moisture content observed in the processed wheat products may be traced to the process conditions employed in their processing. Also, wheat crop is lignocellulosic and may be rich in hemicellulose (the water bearing part of a lignocellulosic plant).

Generally, all the wheat samples were observed to have appreciable amount of crude protein (%) which ranged between 12.28-16.61%. GP1 has the highest % protein content (16.61%) followed by WH4 (15.75%) and SM1 has the lowest percentage crude protein (12.28%). The percentage crude protein values were observed to be slightly higher than the values reported by Goshal *et al.* (2013) of 12.219% when he studied the effect of partially purified xylanase on the quality attribute of whole wheat bread during storage using wheat composites. The difference observed may arise from the nature of samples studied. The high percentage crude protein can be attributed to the improved rheological properties of the wheat flours as it increases the gluten strength of the flour. Although, high crude protein content in form of gluten is known to cause intolerance (coeliac disease) according to Rosell and Segura (2011).

The unprocessed wheat samples were observed to have higher Crude Fiber contents 1.16-1.01% when compared to the processed wheat samples 0.69-0.89%, This can be traced to its multi-layered fiber-rich bran of wheat and thus, serves as the basis for its use as source of food for diabetic patients. It also lowers serum cholesterol level and hypertension (Shewry, 2009). The crude fiber content was observed to be lower than the result reported by Balarabe *et al.* (2017) when he analyzed the proximate composition of bread samples (1.49-6.0%). The reason for the observed difference may be due to the difference in the nature of the samples.

The range of ash content observed was 1.80-0.28%. The unprocessed wheat samples had higher ash contents ranging from 1.59-1.80% when compared to the processed samples (1.28% - 0.28%). WH1 had the highest ash content (1.80%), followed by WH2 (1.72%) while SM1 has the lowest ash content (0.28%). The values of the ash content observed were found to be close to the ash content reported by Balarabe *et al.* (2017) of 1.33 - 3.103%. The slight difference observed may be due to difference in the nature of samples analyzed. The higher ash content observed in the unprocessed sample may be due to the difference in process conditions employed. High ash content indicates corresponding high inorganic mineral contents. The total carbohydrate content (%) of the samples ranged from 70.09 - 64.90%. The unprocessed wheat samples had higher carbohydrate contents ranging from 70.09 - 66.39% when compared to the processed samples (68.80 - 64.90%). WH4 (70.09%) has the highest % carbohydrate contents followed by GP2 (68.80%), while GP1 (64.90%) has the lowest percentage carbohydrate content. However, WH1 (383.95J) was found to value of energy, follows by WH4 (373.38J), while SP1 (329.32J) was found to contain the least energy value. All the wheat samples contain appreciable amount of percentage carbohydrate, making them a rich source of energy for the human system.

4.2 Mineral Contents

Table 4.2. shows the mineral content of unprocessed and processed wheat samples. Their ranges were reported as follows; Na (3.64-6.82 mg/100g), K (161-572 mg/100g), Ca (29.74-56.20 mg/100g), Mg (46.17-92.33 mg/100g), P (128.23-282.20 mg/100g), Fe (2.87-6.58mg/100g) and Zn (2.57-6.75 mg/100g).

In this study, it was observed that K (Potassium) was the most abundant element present in all of the samples. The K content of the unprocessed wheat samples were higher in abundance ranging

from 506 - 572 mg/100g when compared to that of the processed wheat samples 482.50 - 161.17mg/ 100g. This was observed to be lower than the K content reported by Arewa (2017), in the range of 782 - 893mg/100g in an experiment on wheat-potato composite flour. This may be due to the difference in the nature of sample studied. From the results obtained, it was observed that all of the unprocessed wheat samples conformed to the DRI (Direct Reference Intake 470mg/g), while some of the processed wheat samples as (such as GP2 296.97mg, GP3 222.80mg, GP4 353.73mg, SP1 165.30mg, SP2 166.17mg and SP4 161.51) had K-content values below the DRI. Adequate intake of potassium helps in lowering blood pressure, reduce the risk of kidney stone diseases. Inadequate intake of potassium may cause prolonged vomiting, hypokalemia and so on.

Table 4.2: Mineral Contents of the wheat samples (mg/100g)

S/N	Sample	Na	K	Ca	Mg	P	Fe	Zn
1	GP1	4.44±0.05	482.50±0.30	45.84±0.08	63.32±0.12	172.4±07.03	5.82±0.20	4.28±0.19
2	GP2	4.61±0.01	296.97±0.02	52.40±0.01	66.30±0.02	224.6±00.2	6.58±0.01	5.24±0.00
3	GP3	4.23±0.01	222.80±0.50	40.73±0.01	56.33±0.01	268.7±00.02	5.58±0.01	4.70±0.05
4	GP4	4.80±0.00	353.73±0.55	46.27±0.04	61.50±0.04	282.2±00.02	5.90±0.02	4.78±0.02
5	SP1	5.09±0.03	165.30±0.60	30.24±0.02	48.64±0.03	128.23±0.11	3.17±0.03	2.57±0.01
6	SP2	5.20±0.01	166.17±0.02	32.08±0.02	46.17±0.02	144.39±0.01	3.53±0.02	3.81±0.01
7	SP3	3.91±0.01	491.30±0.10	28.36±0.02	59.75±0.04	158.60±0.06	4.30±0.01	5.92±0.02
8	SP4	3.64±0.01	161.57±0.60	27.43±0.01	58.01±0.01	165.38±0.00	3.30±0.01	3.84±0.01
9	WH1	6.35±0.01	506.33±0.30	53.40±0.01	68.64±0.01	271.98±0.01	4.37±0.02	5.19±0.01
10	WH2	5.91±0.01	552.20±0.10	55.77±0.02	84.47±0.01	172.50±0.02	5.18±0.02	5.26±0.03
11	WH3	6.82±0.01	558.35±0.30	50.24±0.02	92.33±0.02	278.32±0.01	4.86±0.01	6.24±0.02
12	WH4	6.61±0.02	572.65±0.01	56.20±0.01	78.44±0.03	156.12±0.03	4.22±0.03	6.43±0.02
13	SM1	4.44±0.11	482.00±4.00	36.77±0.40	63.32±0.03	172.20±0.40	2.87±0.01	4.28±0.05
14	SM2	4.35±0.02	462.03±0.05	29.74±0.01	65.50±0.01	165.85±0.01	3.92±0.02	4.45±0.01
	DRI	1500mg	470mg	100mg	320mg	70mg	18mg	5mg

Key: The values are triplicate determinations \pm standard deviations (SD) for all the samples. GP1-GP4 = general purpose flour, SP1-SP4 = Spaghetti sample, WH1-WH4 = unprocessed wheat sample and SM1-SM2 = semo processed sample.

The amount of P (Phosphorus) content in the samples were observed to be next in abundance to potassium. It ranged from 282.20 - 128.23mg/100g. GP4 (282.20mg/ 100g) had the highest amount of phosphorus followed by WH2 (278.38g/ 100g) and SP4 (128.23mg/ 100g) had the lowest phosphorus content. The result obtained was found to differ from those reported by Roy *et al.* (2015) who evaluated the mineral composition of wheat flour in the critics of Brazil (81 - 107.5mg/100g) and Arewa (2017), who carried out physicochemical evaluation of wheat potato composite flour (38.86mg/100g). This may be due to the difference in the nature of samples studied and the process conditions the samples were subjected to. All the wheat samples were found to contain appreciable amount of phosphorus (higher) when compared to the DRI of phosphorus (70mg). High intake of phosphorus can lead to higher serum parathyroid hormone (PTH) which has an adverse effect on the bone (such as impaired peak bone mass, bone desorption and great or risks of fracture). Low intake of phosphorus can lead to loss of appetite, anemia, muscle weakness, rickets and so on.

The Mg (Magnesium) content was found to be higher in the unprocessed wheat samples which ranged from 68.64-84.74mg/100g when compared to the values of the processed wheat samples (46.17-66.30mg/100g). The magnesium content values observed were higher than that reported by Roy *et al.* (2015) (35mg/100g), this variation may be due to the difference in the samples studied. This was observed to be lower in values when compared to the DRI (320mg). Adequate intake of magnesium aids active transportation of Ca (calcium) and K (Potassium) ions across the cell membrane, conduction of nerve impulses, muscular contractions and so on. Low intake of magnesium may result in gastrointestinal disorder, renal disorder and so on. Excess intake of magnesium may cause increased blood pressure.

The Ca (calcium) content of the wheat samples ranged from 29.74-55.84mg/100g. The unprocessed wheat samples have higher Ca contents 50.24-55.84mg/100g when compared to the processed wheat samples (29.74-52.40mg/100g). This was found to be in disagreement to the Ca content value reported by Arewa, (2017) and Roy *et al.* (2105), of 25.01-32.82mg/100g and 27mg/100g respectively. This may be due to the difference in the samples analyzed. The Ca content result was found to be lower than the prescribed DRI (100mg/day) and thus, proves insufficient. Adequate intake of Ca helps in maintaining healthy skeleton, reduces risk of bone fragility and fracture. Inadequate intake of Ca may cause osteoporosis, increased risk of fracture and unhealthy bone structure. Fe (iron) was found to be higher in the processed wheat flour samples (6.58-5.58mg/100g), followed by the unprocessed wheat flour samples (5.18-4.22mg/100g) and the spaghetti samples had the least Fe content 4.30-3.17mg/100g. The results were found to be lower when compared to the result reported by Arewa (2017), who analyzed wheat-cassava and wheat-potato composites (9.66-5.78mg/100g).

The Fe content of the wheat samples were found to be lower than the prescribed DRI (18mg/day), Hence, they do not contain prescribed amount of Fe. Adequate intake of Fe enhances efficient transportation of oxygen in the blood hemoglobin and generation of cellular energy. Low intake of Fe may cause anemia and high risk of gastrointestinal cancer (65yrs and above). High intake of Fe may increase the risk of type-2-diabetes mellitus (T2DM) according to Wiley *et al.* (2015).

The Na (sodium) content values were observed to be higher in the unprocessed wheat samples (6.82-5.91mg/100g) when compared to the processed wheat samples (5.20-3.64mg/100g). The results were found to be in non-conformity with the DRI (1500mg/day) and do not provide the prescribed DRI sodium intake although a minimum amount of salt is needed for survival. Adequate intake of Na aid proper regulation of concentration and charge differences across cell

membranes, it is important in the absorption of chloride, amino acid, glucose and water. Low intake of Na may not cause any significant effect but the absence of the required amount may result to prolonged vomiting, muscle cramps, nausea and so on. High intake of Na may result to cardiovascular disease in overweight adults (Harper, 2010).

The amount of Zn (zinc) was found to be higher in the unprocessed wheat samples (6.43-5.19mg/100g) when compared to the processed wheat samples (5.92-2.57mg/100g). These results were found to be in close range with those reported by Arewa, (2017) (7.67-7.89mg/100g) though the nature of the sample differs. Most of the samples were observed to contain sufficient DRI (5mg/day). Adequate intake of Zn helps in proper functioning of the immune system, egg fertilization and many enzymatic activities. Low intake of Zn may cause less developed testes and reduced sperm (males) according to Soetan *et al.* (2010).

4.3 Farinograph Analysis

Table 4.2.3 Show the results of the farinographic properties of the unprocessed and processed wheat samples. The ranges of the farinography properties were as follows; D 1:28-6:04 mm:ss, C 495-518 BU, W 56.8-72.7%, S 00:57-4:6 mm:ss, M 17-85 BU, F 33-146 BU and T 3:17-14:37 mm:ss.

Table 4.3: Farinography properties of the wheat samples

S/N	Sample	D(mm:ss)	C(BU)	W(%)	S(mm:ss)	M(BU)	F(BU)	T(mm:ss)
1	GP1	02:04	502	61.5	1:15	85	34	03:25
2	GP2	02:10	504	60.7	00:50	68	33	03:17

3	GP3	01:28	496	60.3	00:57	47	39	03:55
4	GP4	01:50	493	56.8	01:28	52	38	03:48
5	WH1	01:35	509	61.4	00:59	21	133	10:11
6	WH2	02:20	512	62.9	03:43	17	129	08:58
7	WH3	02:07	510	68.9	04:07	19	142	11:36
8	WH4	06:04	518	72.7	04:26	21	146	14:37

Key: D = Dough development time, C = consistency, W = Water absorption capacity, S = Stability time, M = Mixing tolerance index, F = Farinograph quality number and T = Time to breakdown, GP1-GP4 = processed general purpose flour and WH1-WH4 = unprocessed wheat sample.

The non flourly samples (pasta samples) could not undergo farinography measurements due to low/lack of rheological properties. The D (Dough Development Time) for the wheat samples ranged from 01:28 -06:04 mm:ss. The unprocessed wheat flour samples were observed to have higher D ranged (04:07-06:04 mm:ss). This was observed to be in agreement with the farinography properties reported by Ali *et al.* (2015) when he evaluated the effect carbohydrase on the rheology and end qualities of wheat flour cookies (4:00-6:35 mm:ss). The observed difference between the unprocessed and processed samples may be due to the difference in the nature of the unprocessed wheat flour samples. Also, the processed wheat flour samples may have gone through process conditions (both physically and chemically) that may have changed/alter its properties.

C (The dough consistency) of the wheat flour samples range from 495-518BU. WH4 has the highest consistency (518FE) followed by GP2 (514FE) and GP4 (493FE) has the least dough consistency. This range was observed to be higher than the range of dough consistencies reported by Diosi *et al.* (2015) when evaluating the role of farinography and sedimentation on wheat flour qualities (493-485BU). This may be due to the difference in the nature of samples analyzed. When the dough consistency is high, there is corresponding increasing in D (Dough Development Time), and W (Water Absorption Capacity) / F(Farinographic Quality Number) and T (Time Taken to Breakdown). This indicates low baking quality of the flour as consistency towards or equal to 500BU indicates excellent flour. The time taken for the dough and the extent of dough rise among part of the factors influencing the baking quality of the flour.

W (Water Absorption Capacity) is the amount of water expended or absorbed by a flour sample when carrying out a farinography. It is observed to be high in the unprocessed wheat flour samples which ranged from 63.9-72.70% when compared to the processed flour samples 56.8-61.50%. The difference in water absorption capacity maybe due to the difference in the nature of the flour samples experimented. The observed range in the processed samples were observed to be within the range reported by Ahmed *et al.* (2015) after determining the rheological and physicochemical properties of wheat flour samples from Ukraine (57.99-62.23%). High W (Water Absorption Capacity%) influences the D as it takes longer for the dough to be developed which invariably extends it's time to breakdown (T) thereby enhancing high F (farinography quality number), thus, reducing the baking quality of the wheat flour samples as all the aforementioned factors are of positives effects in decreasing values.

The S (stability time) indicates the time difference to the nearest half minutes between the arrival and the departure time. The unprocessed flour samples has higher stability time ranging from 4:26 - 3:44 mm:ss when compared to the processed flour samples (1:2 - 00.50 mm:ss). The stability time range for the processed flour samples were observed to be in close proximity the range reported by Diosi *et al.* (2015) from the evaluation of farinograph properties on wheat samples (1:21-1:06 mm:ss). This difference may be due to difference in the nature of samples analyzed. Low stability time indicates the suitability of flour for baking purposes.

The M (Mixing Tolerance Index) of the flour samples indicates the mixing tolerance of the flour samples. The unprocessed flour samples were observed to have higher M (BU) 85 - 47BU when compared to the processed flour samples (23 - 17BU). The mixing tolerance index (M) for the processed samples was observed to be within the range reported by Ali *et al.* (2015) from the measurement of the rheological properties of wheat flour cookies (16-22BU). This indicates excellent M for the processed flour samples as M greater than 30BU indicates less tolerance and less difficulty in mechanical handling. The difference in M (BU) values may be due to the nature of samples and the process conditions subjected to (for example, the processed flours).

The F (Farinographic Quality Number) is an indicator of farinographic property quality. The unprocessed flour samples have higher F (129 - 146BU) compared to the processed flour samples (38 - 33BU). This was found to be in agreement with values reported by Diosi *et al* (2015) (30-36BU). High F values indicate corresponding high M values and low farinographic properties. The high difference observed between the unprocessed and processed wheat four samples may be due to the nature of samples and the process conditions the processed samples were subjected to.

The T (Time to Break Down) was observed to be higher in the unprocessed flour samples (08:33-14:37 mm:ss) when compared to the processed wheat flour samples (03:17 - 03:48mm:ss). The T measures the strength of a flour sample source. Higher T mm:ss indicates higher M (BU), higher W (%) and higher F (BU).

4.4 Sedimentation

The sedimentation volumes of the wheat samples ranged from 12-33 cm³. Processed wheat samples had higher sedimentation volume ranging from 24-33 cm³, when compared to the unprocessed wheat samples whose volume ranged from 12-14 cm³.

Table 4.4: Sedimentation Volume of the Wheat Samples

S/N	Sample	Sedimentation(cm ³)
1	GP1	33
2	GP2	33
3	GP3	31
4	GP4	32
5	WH1	14
6	WH2	13
7	WH3	14
8	WH4	12
9	SM1	25
10	SM2	24

Keys: GP1-GP4 = processed general purpose flour, WH1-WH4= unprocessed wheat sample and SM1-SM2 = processed semo sample.

It was observed that spaghetti samples could not undergo this analysis because they were not properly dispersed in the sedimentation liquid due to their sample nature. The processed flour samples have higher sedimentation volume ranging from 31- 33 cm³ which agrees to the sedimentation volume range reported by Ali *et al.* (2015) (31-35cm³). This is then followed by the semo samples (24 - 25 cm³) and the unprocessed wheat flour samples had the lowest sedimentation volume ranges (12 – 14 cm³). High sedimentation volume indicates strong gluten

properties and possibility for adulteration while low sedimentation volume indicates weak gluten properties and low possibility for adulteration. These findings further explain why the processed flours are better in the baking industry compared to the unprocessed flours as high gluten strength enhances the rheological properties of the flour during dough formation and the bread volume after baking.

4.5 Chemometrics

All the data gotten from proximate compositions, mineral contents, sedimentation and farinograph analysis were subjected to chemometrics to obtain more information on existing disparities among samples, enhance human identification and pattern recognition. PCA and HCA were the techniques employed.

1.5.1 Principal component analysis (PCA)

1.5.2 Eigen value plots

The Eigen value plots determine the selection of the principal components (PCs) that will help capture the major variables responsible for the sample attributes.

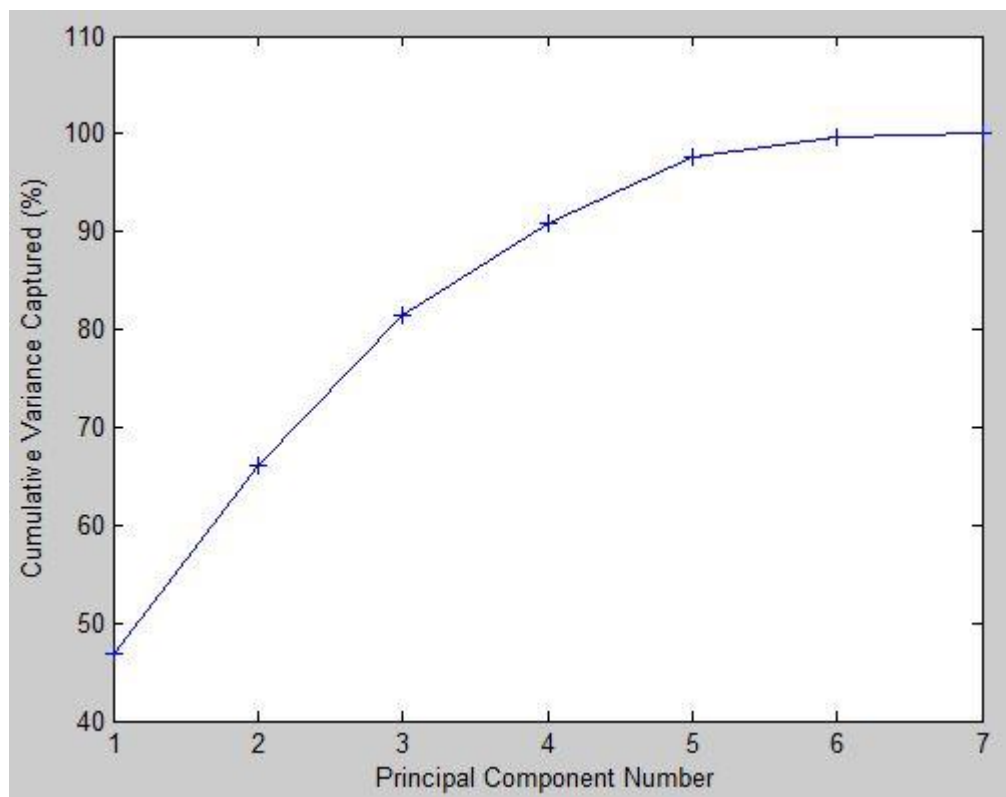


Figure 4.1: PCA Eigen value plot for proximate composition

The plot above shows a cumulative variance capture between 50 and 70% on the x-axis and principal component number between 1.5 and 2.3 on the y-axis which captures the majority of sample variation or similarity.

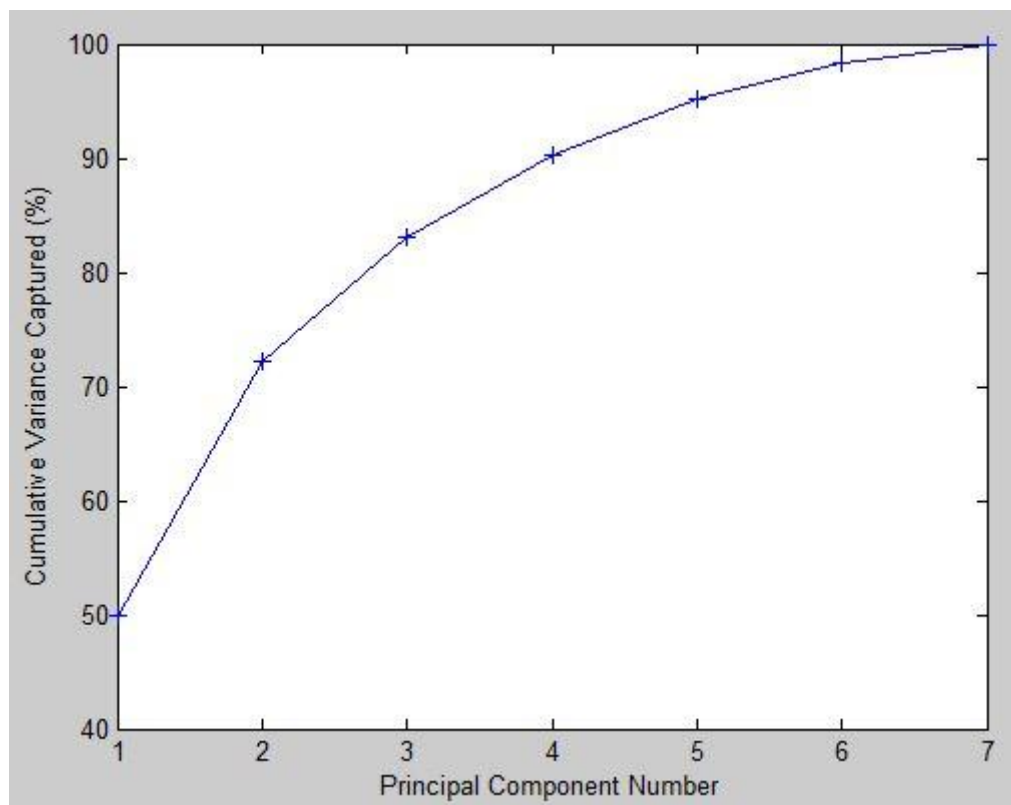


Figure 4.1.1: PCA Eigen value plot for mineral contents

The Eigen value plot shows a cumulative variance capture of the samples at PC 50% and 90% which is enough to capture all the information regarding variation, similarities and close relationship existing amidst the samples and their principal components.

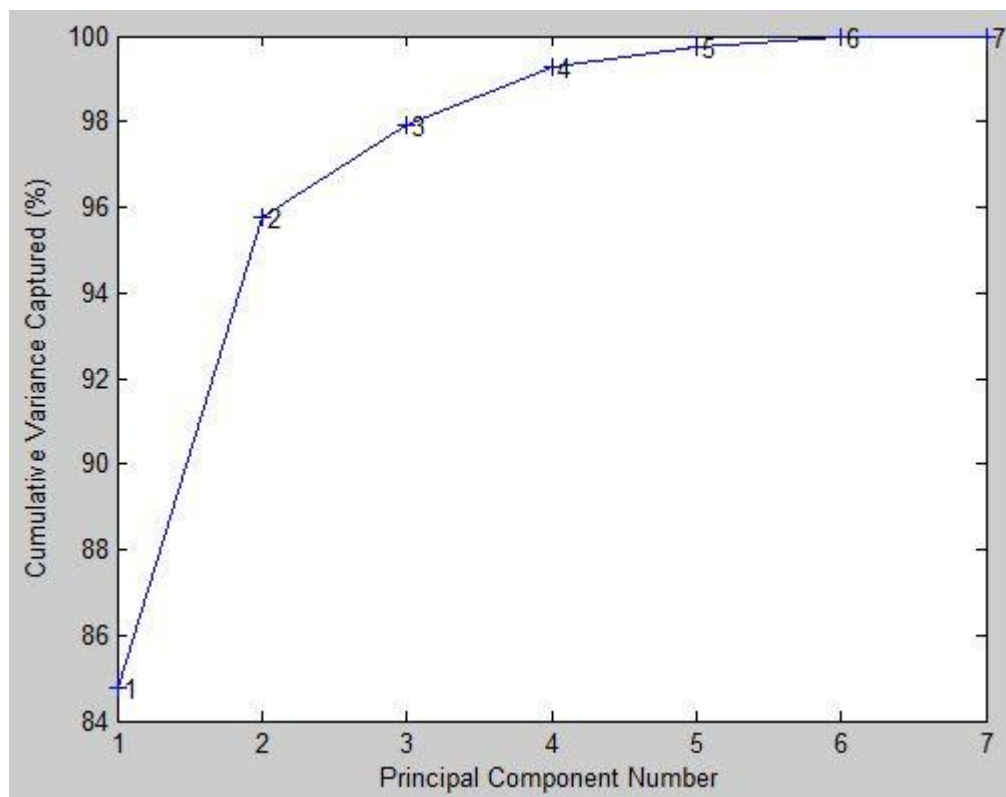
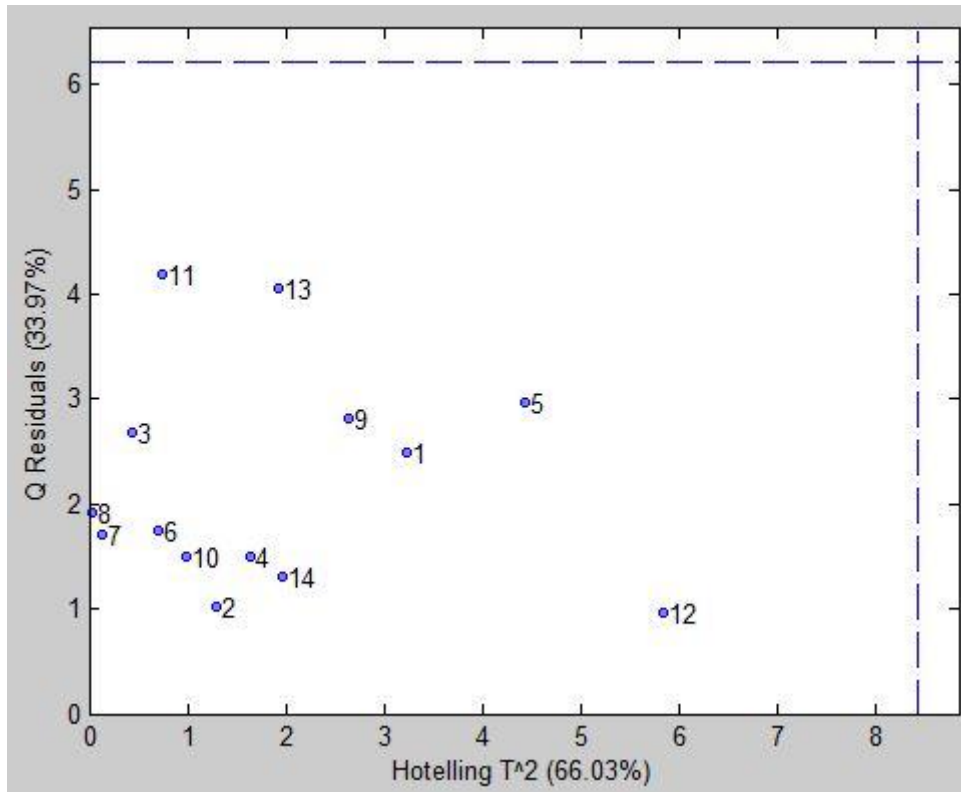


Figure 4.1.2: Eigen value plot for farinography properties

The Eigen value plot shows a cumulative variance capture of the samples at PC 50% and 90% which is enough to capture all the information regarding variation, similarities and close relationship existing amidst the samples and their Principal components.

1.5.3 PCA Hotelling T^2 / Q residual plots

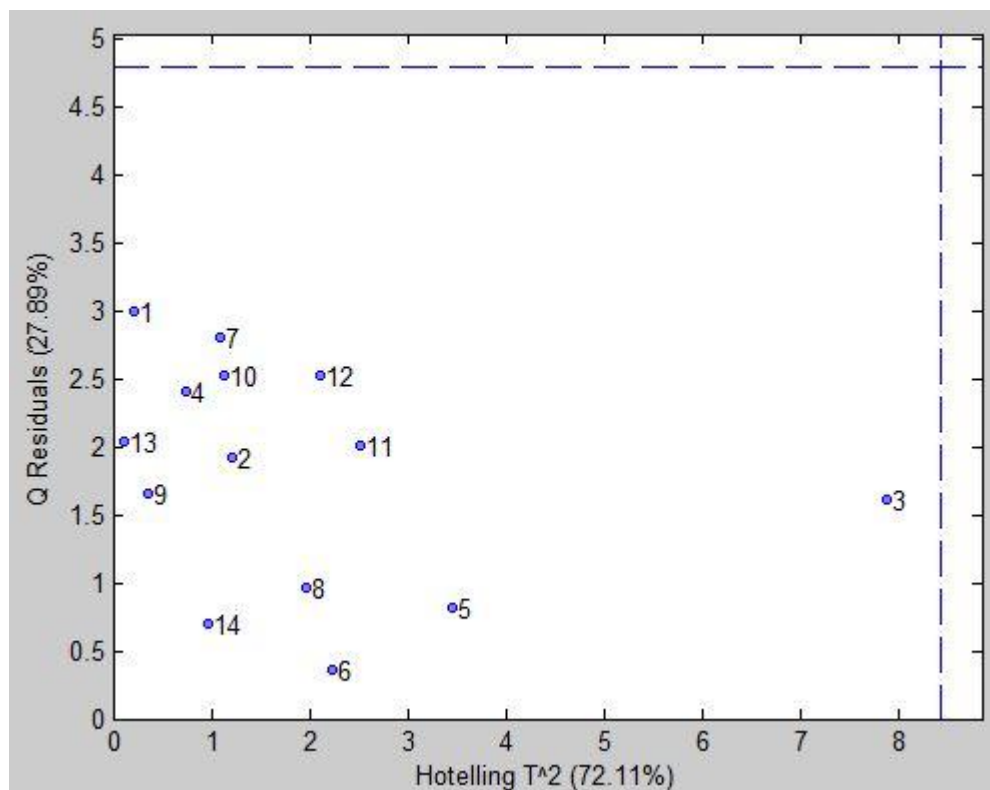
The Hotelling T^2 plots capture significant multi-variate distances which reveal or explains the reason for exceptional behavior of a sample.



Key: 1 to 4 = processed general purpose flour, 5 to 8 = processed spaghetti samples, 9 to 12 = unprocessed samples and 13 and 14 = processed semo samples.

Figure 4.2: PCA hotelling T^2 plot for proximate composition

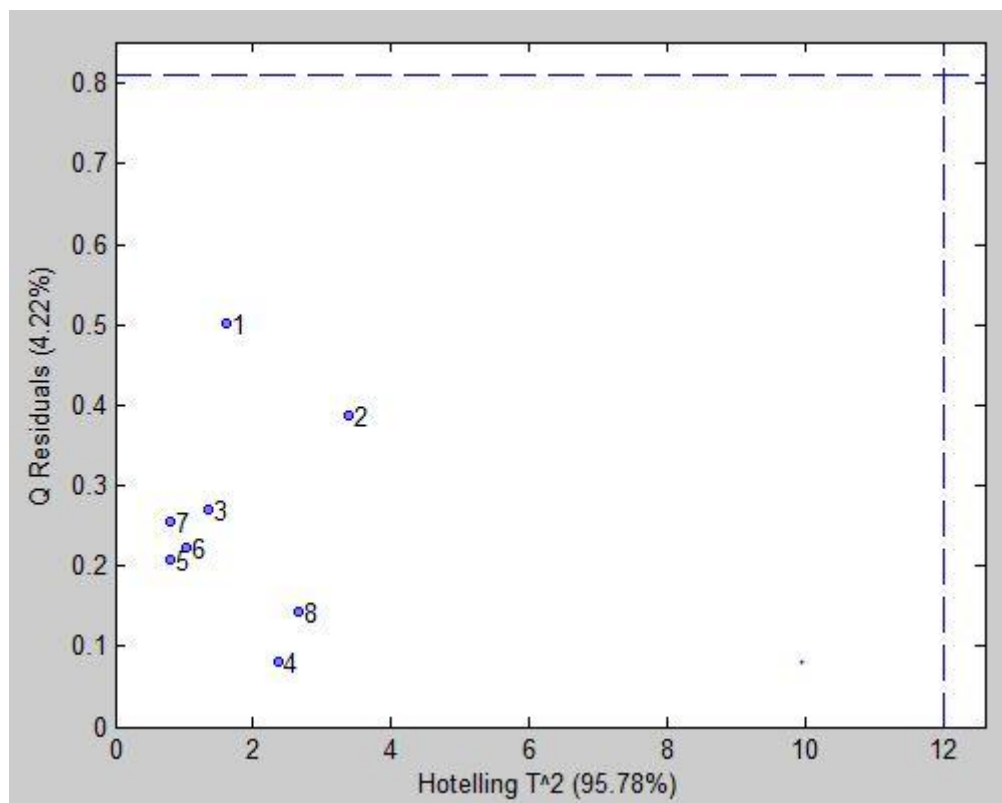
This plot shows a cumulative variance captures of 33.97% and 66.03% (PC1) which explains for any observed uniqueness. From the plot, samples with pronounce variation are seen to be far away from the origin. Thus, sample 12(WH4) which is one of the unprocessed wheat products was observed to have the most pronounced difference in terms of proximate content compared to the rest, this may be due its principal constituents as shown by its proximate properties from its table.



Key: 1 to 4 = processed general purpose flour, 5 to 8 = processed spaghetti samples, 9 to 12 = unprocessed samples and 13 and 14 = processed semo samples.

Figure 4.2.1: PCA hotelling T² plot for mineral contents

This plot shows a cumulative variance captures of 27.89% (PC2) and 72.11% (PC1) which explains for any observed uniqueness. From the plot, samples with pronounce variation are seen to be far away from the origin. Thus, sample 3 (GP3) which is one of the processed wheat products (general purpose flour) is observed to have the most pronounced difference in terms of mineral content compared to the rest as earlier revealed by the score plot, this may be due its principal constituents as shown by its mineral contents as extracted from its table.



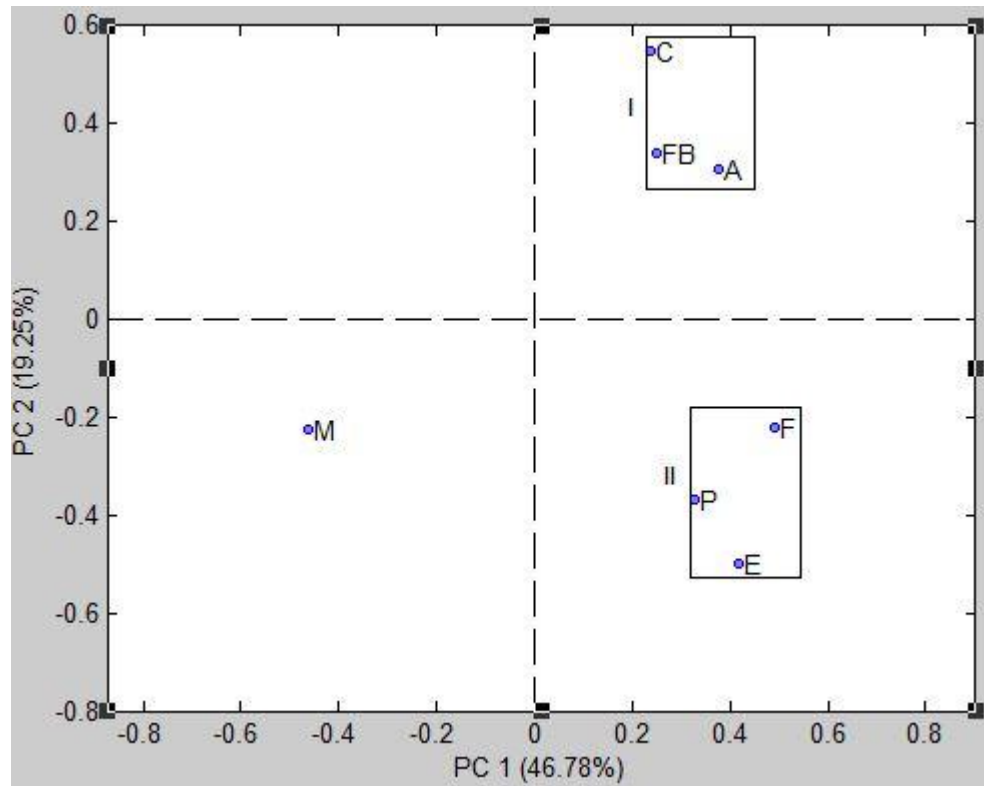
Key: 1 to 4 = processed general purpose samples and 5 to 8 = unprocessed wheat samples.

Figure 4.2.2: hotelling T² plot for farinography properties

The plot shows cumulative variance capture at PC2 (4.22%) and PC1(95.78%). It was observed that samples of common farinographic properties concentrates towards the origin. It is seen that the samples 1 (GP1) and 2 (GP2) show unusual farinographic properties which may be due to difference in their processing techniques or additives (such as gluten) employed. As the presence of gluten helps in improving Dough Development Time (D), Consistency (C) and Mixing Tolerance Index(M). Only Figure 4.5.2.1 showed a close cumulative variance capture when compared to the report drawn from Adebisi *et al.* (2017) PC1 (31.13%) and PC2 (66.0%), this may be due to the difference in the data sets analyzed.

4.5.4 PCA Loading Plots

The loading plots reveal the informations regarding the principal components responsible for the variation among samples.



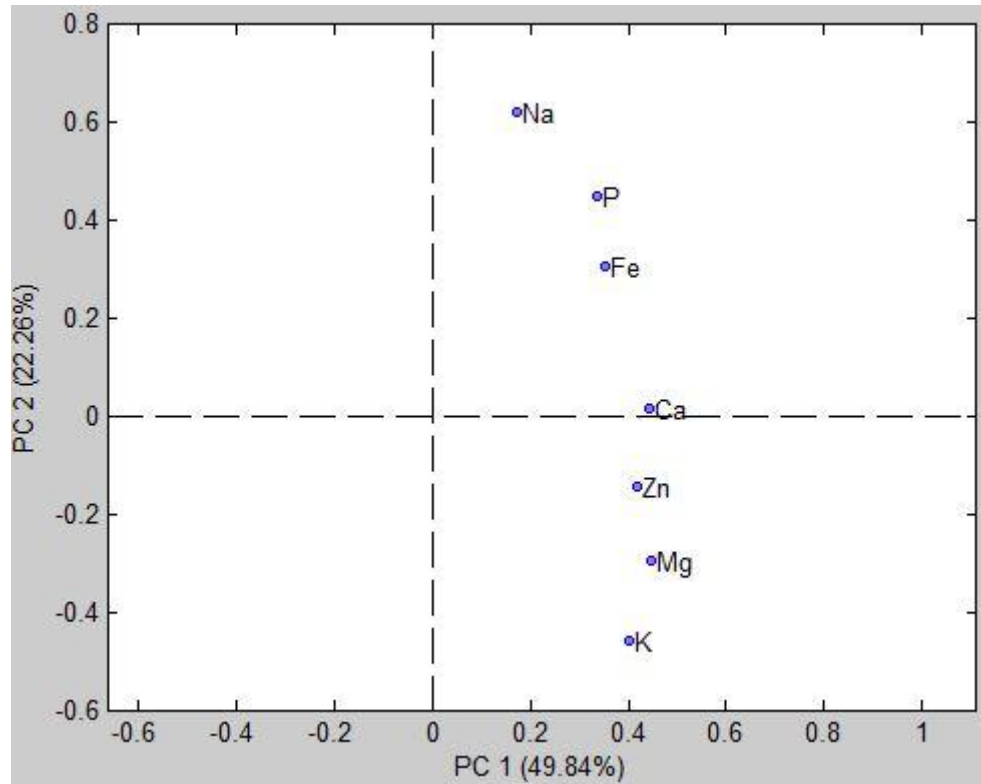
Keys: M = moisture, C = carbohydrate, FB = crude fibre, A = ash, P = crude protein, F = fat and E = energy.

Figure 4.3: PCA loading plot for proximate composition

The loading plot shows a cumulative variance capture of 19.25% (PC2) and 46.78% (PC1) which accounts for the dominance of the respective principal component observed.

The loading plots shows that group 1 which contain members C (carbohydrate), FB (crude fibre) and A (Ash) are the dominant variables or constituents that makes up all of the samples as they are well pronounced in PC1 and PC2 are positive axis. This report was found to agree with the report made by Kibar (2019) who assessed the proximate composition of de-hulled eikorn wheat samples at varying moisture contents, carbohydrate and crude fiber were the major principal

components observed at 54.48% (PC2) and 35.72% (PC1). In PC2, it observed that variables such as F (fat), P (protein) and E (energy) only show dominance in PC2 axis which makes members of group II less influential principal components compared to those of group I. M (moisture) which is found on the axis of PC2 shows less or insignificant influence on the principal component of the samples.

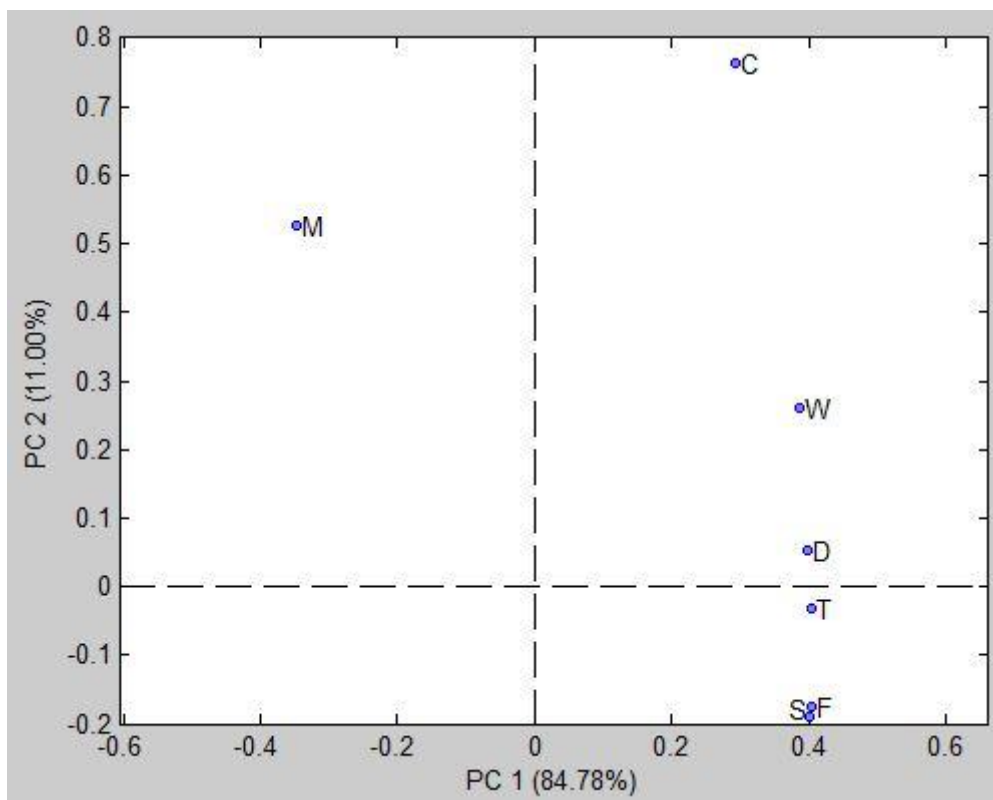


Keys: Na = sodium, P = phosphorus, Fe = iron, Ca = calcium, Zn = zinc, Mg = magnesium, and K = potassium.

Figure 4.3.1: PCA loading plot for mineral contents

The Loading plot shows a cumulative variance capture at 22.26% (PC2) and 49.84% (PC1) which gives the information regarding the observed principal components. Na, P and Fe are the dominant principal components that influence the properties of most of the samples as they show positive correlation in both PC1 and PC2 axis. Although, this was found to disagree with the principal component reported by Wang *et al.* (2020) who carried out PCA on the data sets

obtained from mineral contents of wheat varieties which revealed (K) as the major principal component cumulative variance capture of 21.25%(PC2) and 69.33%(PC1). Zn, Mg and K are less dominant as they show positive correlation only in PC2 axis. Hence, has lesser influence on the properties of the samples. Ca has no significant contribution to either of the PC axis. Hence, it is not a principal component and does not have significant influence on the properties exhibited by the samples.



Keys: M = mixing tolerance index, C = dough consistency, W = water absorption capacity, D = dough development time, T = time to breakdown, S = stability time and F = farinograph quality number.

Figure 4.3.2: Loading plot for farinography properties

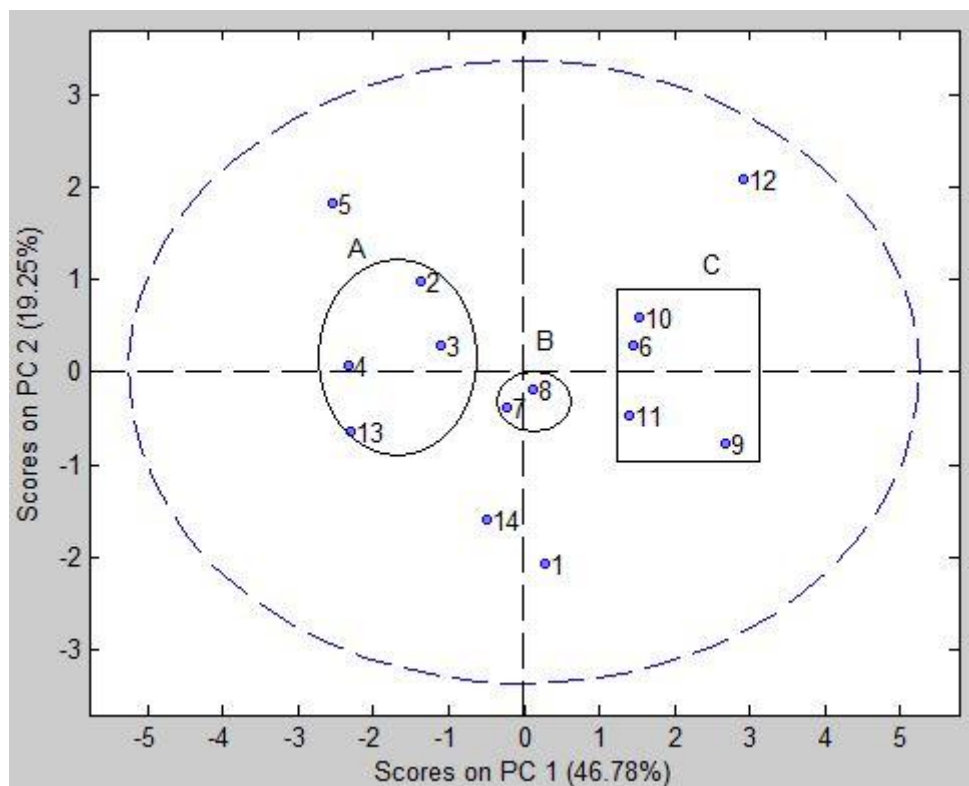
The loading plot shows a cumulative variance capture at PC2 (11.00%) and PC1 (84.78%). C (consistency), W (water Absorption capacity) and D (Dough development time) are the major

principal components factors as they are positively correlated in PC1 and PC2). C has the most pronounced positive value as the key principal component. C is the most dominant factor that defines if a sample can be measured farinographically according to literature as non-dough forming wheat samples cannot undergo farinographic test. W (water absorption capacity) is the next dominant principal component that contributes to farinographic properties of the samples. The water absorption capacity of a flour sample has effect on the rheological properties of the dough. D (Dough development time) is least major principal component contributing to the farinographic properties of the samples. T (Time to breakdown), S(speed) and F (farinographic quality number) and less dominant principal components factors that defines the farinographic properties as they have negative correlating values. M (Mixing Tolerance Index) is less dominant when describing farinographic properties as it only has positive correlation in PC1.

4.5.5 PCA Score Plots

The Score plots highlights information about patterns existing in the samples. Thus, reducing the difficulty in visualizing the disparities existing among the samples analyzed.

The two PCs show cumulative percentage variance of 19.25% (PC2) and 47.78% (PC1) which captures the similarities and variations existing among the samples. Groups A, B, C are clearly distinguished.

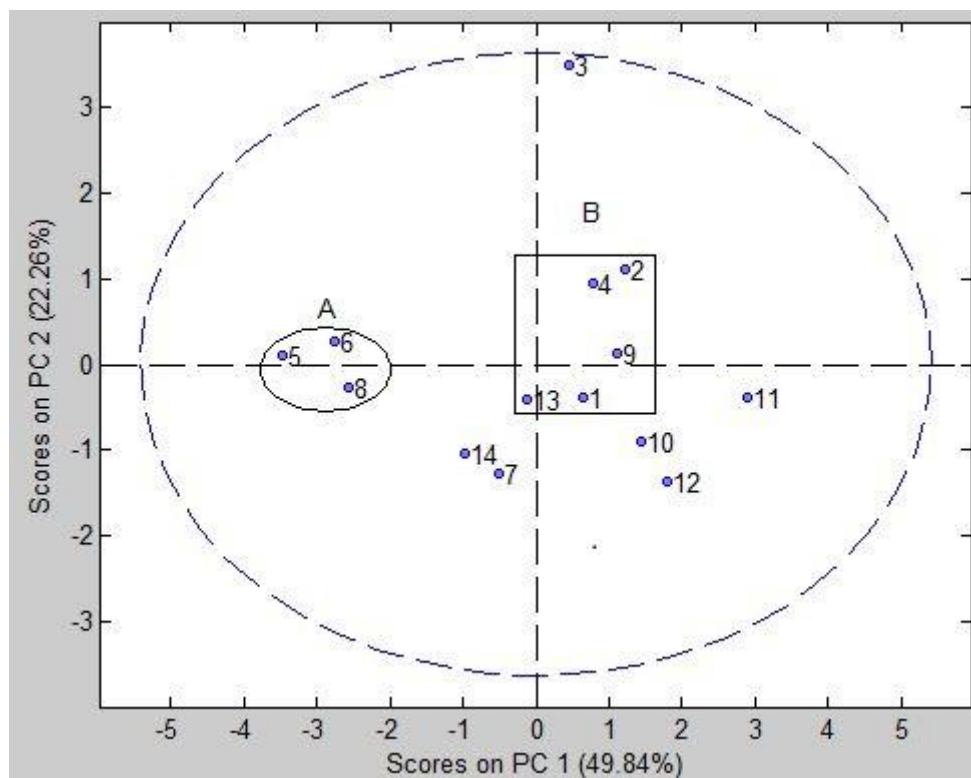


Keys: 1 to 4 = processed general purpose flour, 5 to 8 = processed spaghetti samples, 9 to 12 = unprocessed wheat samples and 13 and 14 = processed semo samples.

Figure 4.4: PCA Score plot for proximate composition

Group A has four correlating members/samples which are 2, 3, 4 and 13. All the four correlating members are observed to wheat flour and semo products, although 2, 3 and 4 are processed wheat flour samples, while 13 is a processed semo samples. The reason for their correlation can be attributed to their close proximity in proximate content values shown in the order of crude fat(%), moisture(%), crude protein(%), crude fiber, ash(%), carbohydrate(%) and energy(J). Sample 2 (GP 2: 3.59 ± 0.11 , 11.83 ± 0.03 , 13.9 ± 0.04 , 0.89 ± 0.02 , 0.71 ± 0.01 , 68.80 ± 0.01 and 355.45), Sample 3 (GP3: 3.41 ± 0.02 , 12.47 ± 0.02 , 15.37 ± 0.11 , 0.73 ± 0.02 , 1.08 ± 0.10 , 66.72 and 343.79), Sample 3 (GP4: 2.87 ± 0.08 , 13.74 ± 0.04 , 14.91 ± 0.04 , 0.82 ± 0.01 , 0.92 ± 0.01 , 66.83 ± 0.04 and 343.79) and Sample 13 (SM1: 4.64 ± 0.04 , 13.02 ± 0.03 , 12.25 ± 0.05 , 0.72 ± 0.04 , 0.28 ± 0.02 , 66.81 and 351.54). Also, their correlating property is seen to cut across both PC1 and PC2 which

implies appreciable source of proximate content in group B contain two members 7, 8 which are observed to be spaghetti samples (SP3 and SP4) and also show their close proximity in proximate content as shown below in the order of crude fat(%), moisture(%), crude protein(%), crude fiber, ash(%), carbohydrate(%) and energy(J). Sample 7 (SP3: 4.62 ± 0.10 , 10.65 ± 0.08 , 15.71 ± 0.03 , 0.88 ± 0.02 , 0.48 ± 0.01 , 67.87 ± 0.02 and 364.93) and Sample 8 (SP4: 4.1 ± 0.04 , 9.80 ± 0.01 , 15.75 ± 0.03 , 0.75 ± 0.20 , 0.82 ± 0.00 , 68.27 ± 0.01 and 366.59). 7 and 8 are seen to be good source of calorie-containing foods, Group C consist of four members 9, 10, 11 and 6 which are raw wheat samples (that is WH1, WH2 and WH3 respectively) while 6 is a pasta sample (SP2). Group C members show closeness in proximate content values as shown below in the order of crude fat(%), moisture(%), crude protein(%), crude fiber, ash(%), carbohydrate(%) and energy(J). Sample 9 (WH1: 6.92 ± 0.04 , 8.19 ± 0.08 , 15.70 ± 0.05 , 1.01 ± 0.04 , 1.80 ± 0.01 , 67.84 ± 0.03 and 383.95), Sample 10 (WH2: 5.59 ± 0.02 , 10.26 ± 0.03 , 14.80 ± 0.06 , 1.05 ± 0.02 , 1.76 ± 0.01 , 67.86 ± 0.01 and 370.85), Sample 11 (WH3 : 6.19 ± 0.03 , 11.00 ± 0.05 , 14.91 ± 0.05 , 1.13 ± 0.02 , 1.44 ± 0.01 , 66.39 ± 0.09 and 369.71) and Sample 6 (SP2: 5.80 ± 0.03 , 8.83 ± 0.11 , 14.80 ± 0.00 , 0.81 ± 0.02 , 1.21 ± 0.00 , 9.03 ± 0.05 and 377.26). Group C members are observed to possess appreciable amount of calorific contents.

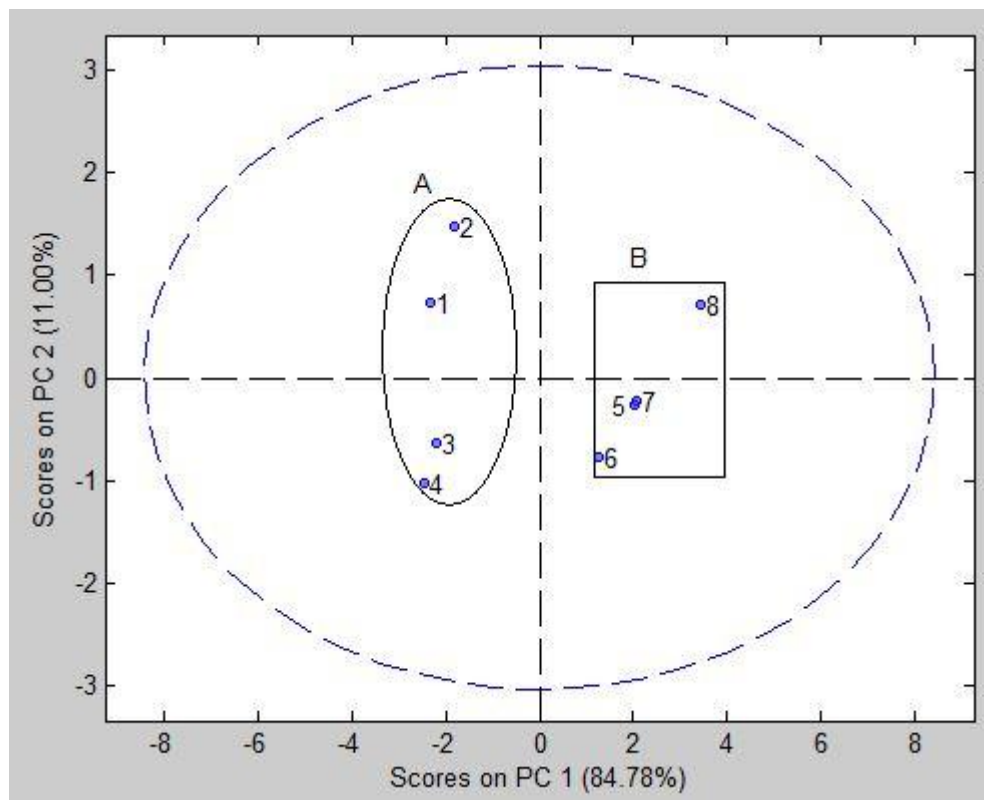


Keys: 1 to 4 = processed general purpose flour, 5 to 8 = processed spaghetti samples, 9 to 12 = unprocessed samples and 13 and 14 = processed semo samples.

Figure 4.4.1: PCA score plot for mineral contents

The score plot shows cumulative percentage variance of PC2 (22.26%) and PC1 (49.84%) which captures the variations and differences existing among samples. Groups A and B are clearly revealed by this plot. Group A contains numbers 5 (SP1), 6(SP2) and 8 (SP4) which are all spaghetti samples (pasta). Group B members are majorly wheat flour samples 1(GP1), 2(GP2), 3(GP3), 4(GP4), 9(WH1) except for 13(SM1) which is a semo sample. The Score plot was able to the group the samples according to the nature of their products. Also, the difference in nature between groups A and B may be due to the difference in process conditions undergone. Sample 13 despite being uncommon to the group B members yet has close mineral content values. This is one of the hidden relations conventional statistics may not reveal easily. Sample 3 (GPF3) has no

significant relationship with the two groups A and B. This may be due to the difference in constitutional make up or product brand.



Keys: 1 to 4 = processed flour samples and 5 to 8 = unprocessed wheat samples.

Figure 4.4.2: Score plot for farinography properties

The score plot shows a cumulative 90% variance capture at PC2 (11.00%) and PC1 (84.78%).

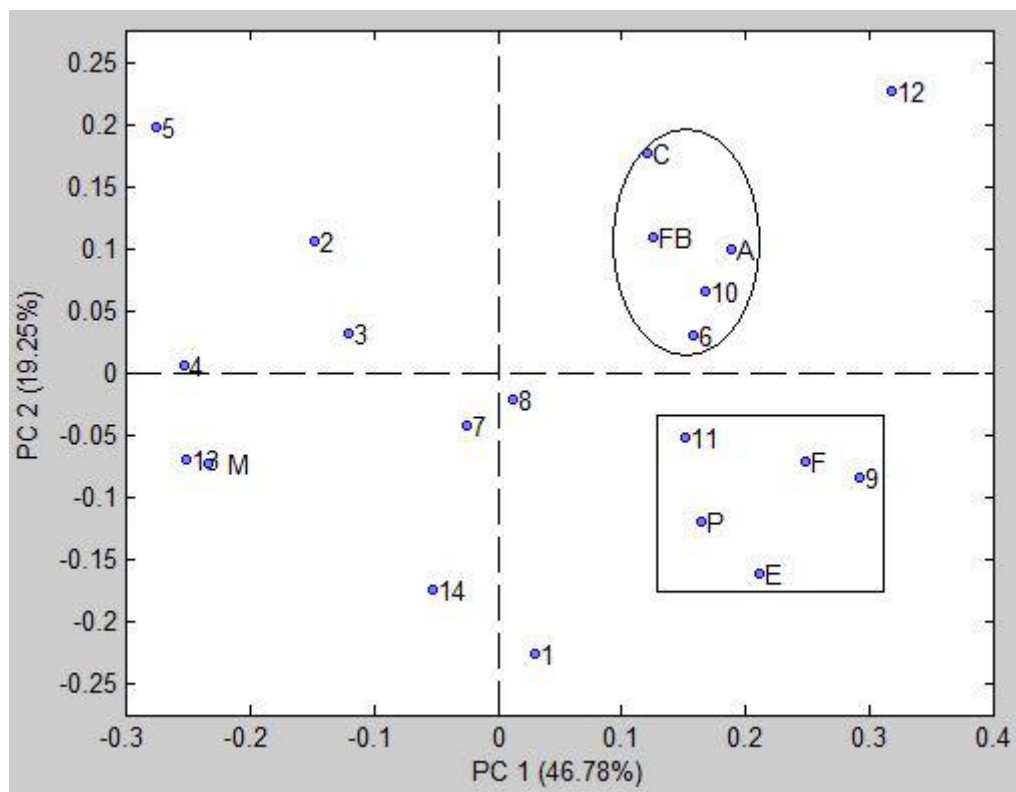
The two visible groups A and B and are distinguished into unprocessed wheat flour samples (B) and processed wheat four samples (A). Group A contains members 1 (GP1), 2 (GP2), 3 (GP3) and 4 (GP4) which are all processed wheat flours samples. Although 1, 2, 3 and 4 are all in the same group but they show negative correlation as they differ in farinographic properties (1, 2, 3 and 4). The negative correlation may be due to Brand/Process differences. Group B is made up of members 5(WH1), 6(WH2), 7(WH3) and 8(WH4) are all unprocessed wheat flours samples. Although, members 5, 6, 7 and 8 are in the same group but they differ in farinography properties

as they are negatively correlated (5, 6, 7 are negatively correlated to 8). The negative correlation may be due to difference in the raw wheat origins.

4.5.6 PCA biplots

The biplot reveal a combine information about samples from both score and loading plots simultaneously, this in turn display correlation between variable(s) and principal component(s).

Also, it enhances sub-grouping of the studied samples into various categories.

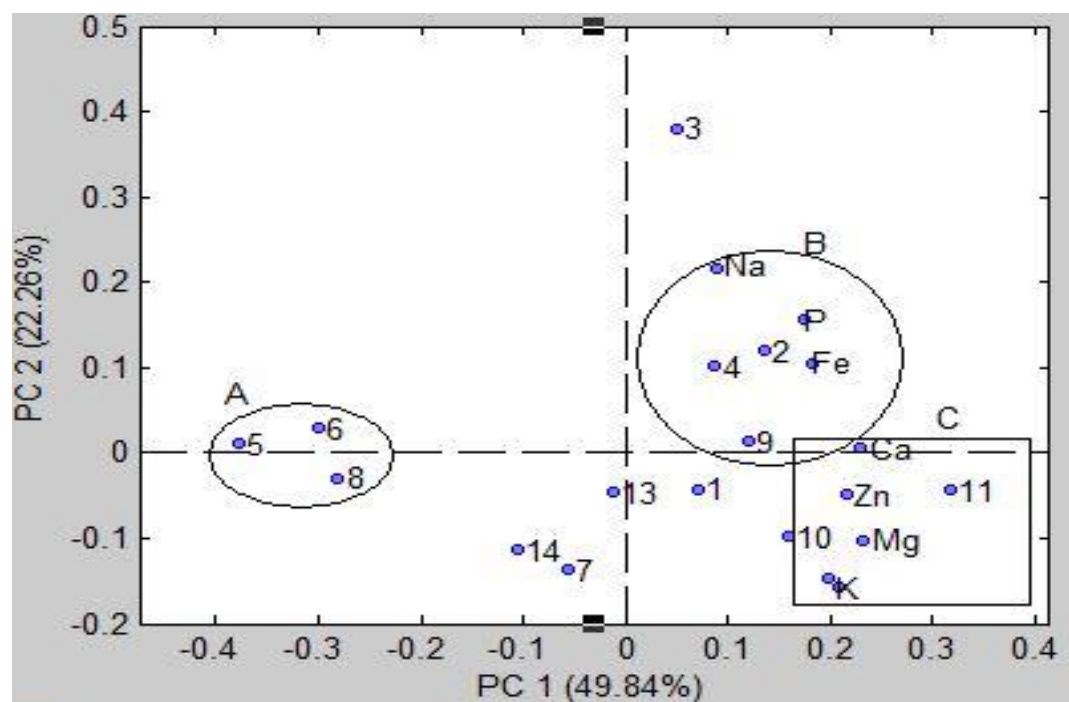


Key: 1 to 4 = processed general purpose flour, 5 to 8 = processed spaghetti samples, 9 to 12 = unprocessed samples and 13 and 14 = processed semo samples, M = moisture, C = carbohydrate, FB = crude fibre, A = ash, F = fat, P = crude protein and E = energy.

Figure 4.5.: PCA Biplot plot for proximate composition

The bi-plot captured two visible groups based on their nutritional content correlation. Group 1 (sphere shaped) contains member 6 (SP2) and 10 (WH1) which are made up of spaghetti sample

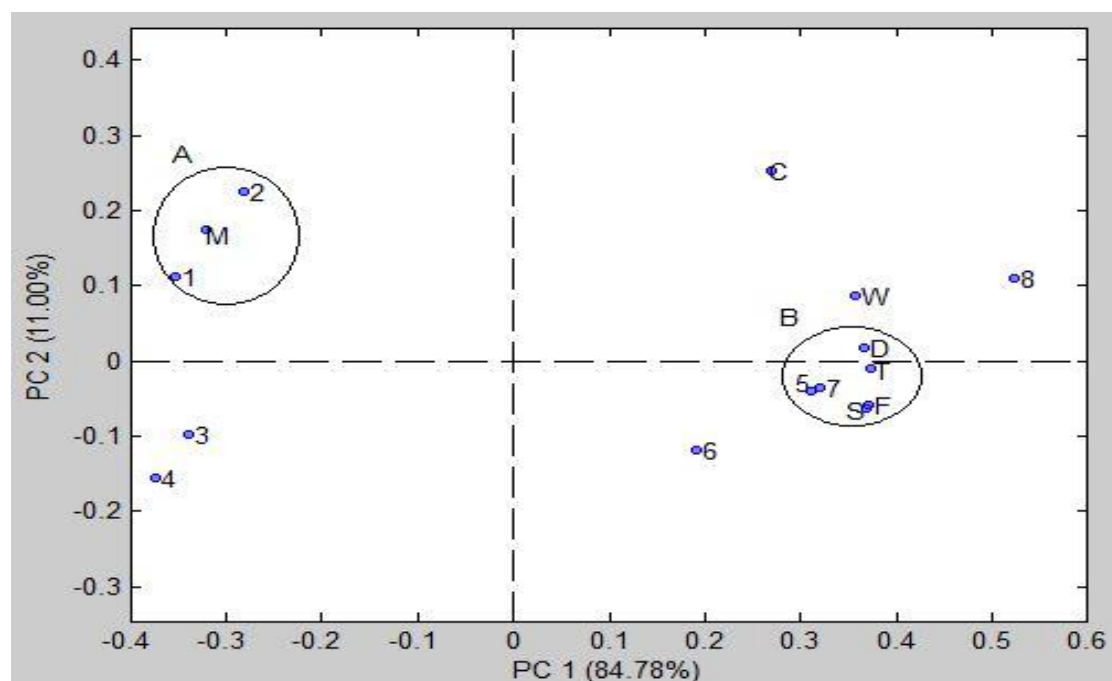
and an unprocessed wheat flour sample respectively. It is observed that 6 & 10 are good source of ash, crude fiber and carbohydrate as revealed by their proximate content values which are shown below in the other; Ash (%), Crude Fiber (%) and Carbohydrate (%). Sample 6 (SP2 : 1.21 ± 0.00 , 0.81 ± 0.02 and 69.03) and Sample 10 (WH2 : 1.76 ± 0.01 , 1.05 ± 0.02 , 67.86). The second group (rectangular shape) comprising of sample 9(WH1) and 11(WH3) are unprocessed wheat flour samples. Sample 11 and 9 are energy giving foods as they are characterized appreciable amount of fat, energy and protein as show by their extracted proximate content values. Sample 13(SM1) which is a processed wheat semo sample is characterized by moisture samples without alphabets show less appreciable amount of macro-nutrients. Contrary to the findings reported by Adebisi *et al.* (2017) having performed PCA on the proximate compositions of cereal grains, reported that none of the sample analyzed were characterized by the principal component (crude protein).



Keys: 1 to 4 = processed general purpose flour, 5 to 8 = processed spaghetti samples, 9 to 12 = unprocessed samples and 13 and 14 = processed semo samples.

Figure 4.5.1: Biplot for mineral contents

The bi-plot provides multivariate information by combining information from the score plot and loading plots. Thus, giving more information compared to the earlier aforementioned plots. The bi-plot captured two visible groups based on their nutritional content correlation. Group 1 (sphere shaped) contains member 6(SP2) and 10(WH1) which are made up of spaghetti sample and an unprocessed wheat flour sample respectively. It is observed that 6 & 10 are good source of ash, crude fiber and carbohydrate as revealed by their proximate content values which are shown below in the other; ash(%), crude fiber (%) and carbohydrate(%). Sample 6 (SP2: 1.21 ± 0.00 , 0.81 ± 0.02 and 69.03) and Sample 10 (WH2: 1.76 ± 0.01 , 1.05 ± 0.02 , 67.86). The second group (rectangular shape) comprising of sample 9(WH1) and 11(WH3) are unprocessed wheat flour samples. Sample 11 and 9 are energy giving foods as they are characterized appreciable amount of fat, energy and protein as show by their extracted proximate content values. Sample 13(SM1) which is a processed wheat semo sample is characterized by moisture samples without alphabets show less appreciable amount of macro-nutrients.



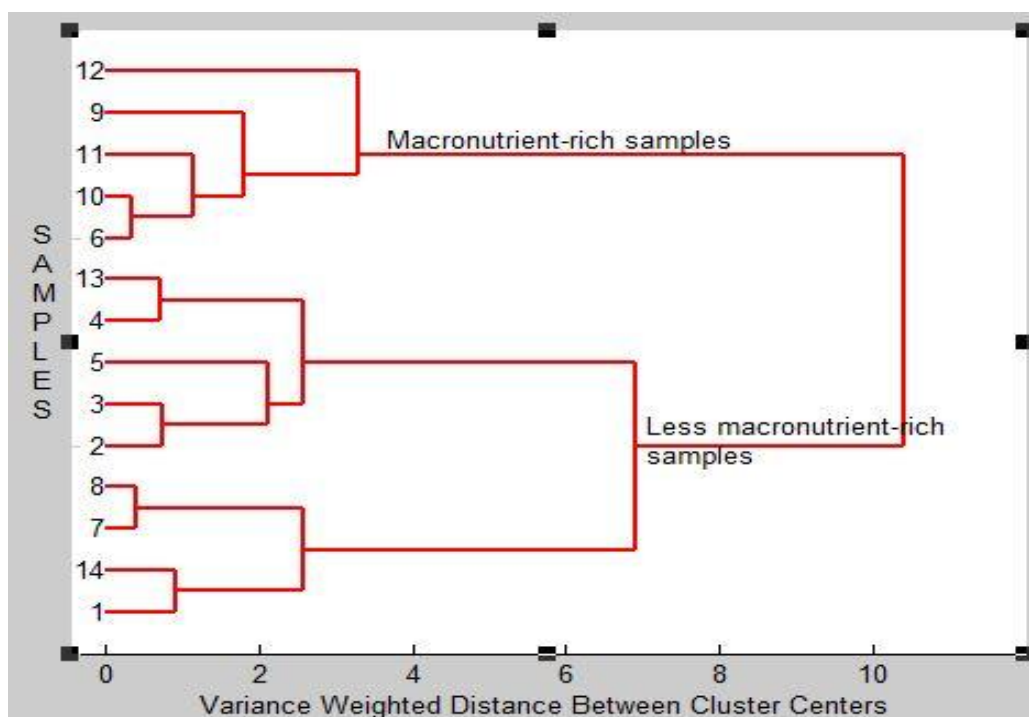
Key: 1 to 4 = processed general purpose flour, 5 to 8 = unprocessed samples, M = mixing tolerance index, C = dough consistency, W = water absorption capacity, D = dough development time, T = time to breakdown, S = stability time and F = farinograph quality number.

Figure 4.5.2: Biplot for farinography properties

The bi-plot shows a cumulative variance capture at 11.00% (PC2) and 84.78% (PC1). The plot shows that none of the samples is characterized by C (consistency) as the standard dough consistency is 500BU. Group A Samples 1 (GP1) and 2 (GP2) can be characterized by M (Mixing Tolerance Index). Group B samples 5(WH1) and 7(WH2) are characterized by D (Dough Development Time), T (Time to breakdown), S (speed) and F (farinography quality number). Samples of 4(GP4) (GP3) shows negative correlation to the major principal components as they are observed to have lower values of the principal components.

4.6. Hierarchical Cluster Analysis (HCA)

The Hierarchical cluster is known for revealing the degree of substitutability of the analyzed samples. It also helps to group samples according to similarity properties such as brands, origin and so on.

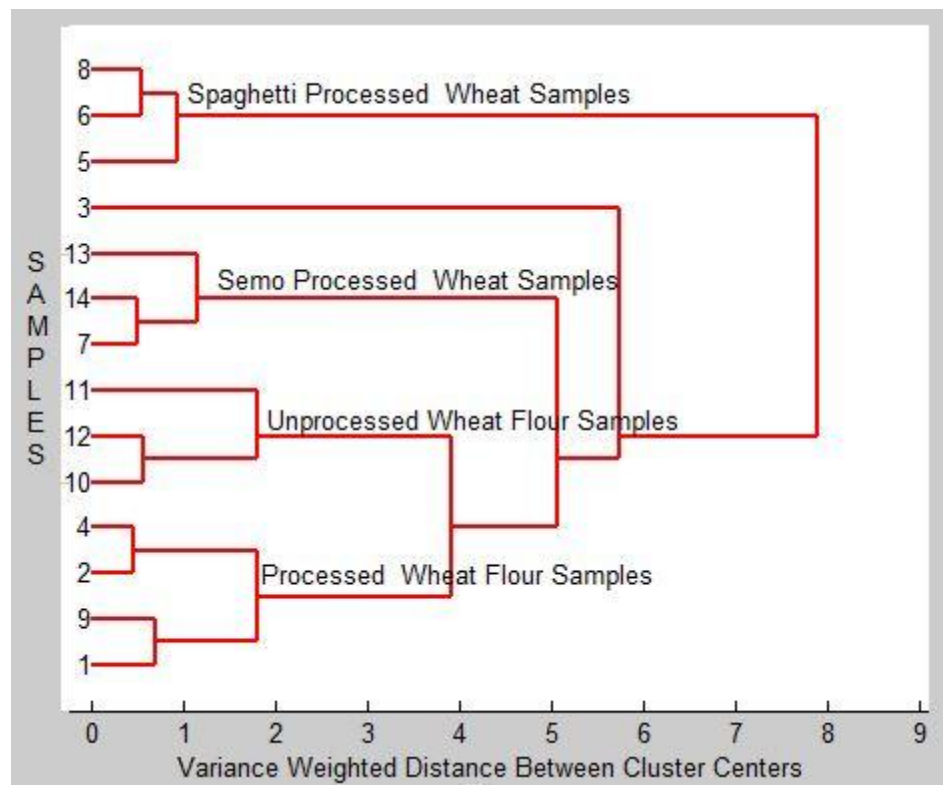


Keys: 1 to 4 = processed general purpose flour, 5 to 8 = processed spaghetti samples, 9 to 12 = unprocessed samples and 13 and 14 = processed semo samples.

Figure 4.6.1 Hierarchical cluster analysis (HCA) result for proximate composition

The clustering of proximate compositions of the 14 wheat samples is presented in the classification was based on the similarities and substitutability of the 14 wheat samples for the six proximate variables. The HCA dendrogram shows three major clusters which reveal the substitutability of wheat samples based on variance weighted distance between cluster centers in the decreasing order of 10 and 6 > 7 and 8 > 4 and 13 > 2 and 3 > 1 and 14 according to their proximate content values in clusters (groups) and sub clusters (close substitutes). The samples are grouped into macro nutrient-rich samples (6,10,11,9,12) and less macro nutrient-rich samples (7,8,2,3,4,13,1,14, and 5) by the HCA dendrogram. This was found to agree with the Kibar (2019) whose HCA report also categorized de-hulled eikorn wheat flour samples at varying moisture content into macronutrient-rich and less macronutrient-rich.

The Hierarchical Cluster Analysis (HCA) dendrogram of the mineral contents of the unprocessed and processed wheat samples reveals substitutability among the wheat samples.



Keys: 1 to 4 = processed general purpose flour, 5 to 8 = processed spaghetti samples, 9 to 12 = unprocessed samples and 13 and 14 = processed semo samples

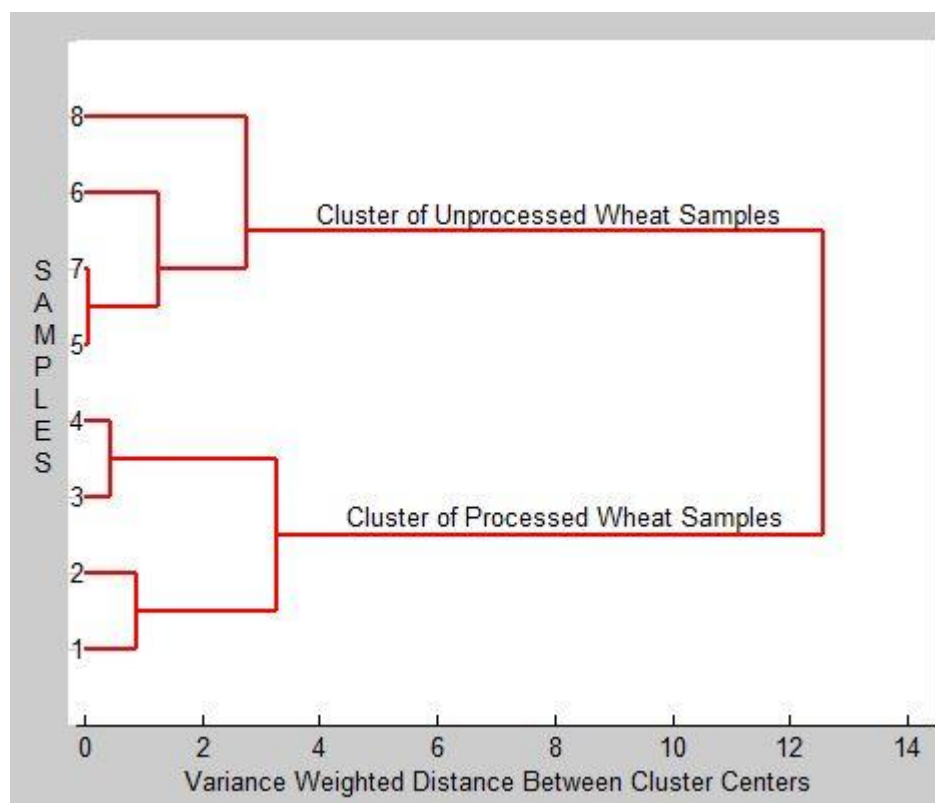
Figure 4.6.2 Hierarchical cluster analysis (HCA) result for mineral contents

Fig. 4.6.2. show four major clusters which reveals the substitutability of the wheat samples according to mineral contents based on Variance Weighted Distance between Cluster Centers and in the decreasing order of 4 and 2 > 14 and 7 > 8 and 6 > 10 and 12 > 1 and 9. This was contrary to findings reported by Wang *et al.* (2020) who carried out HCA on wheat flour varieties and reported that all the samples analyzed were substitutable. This information will aid the consumer in making informed choices. The samples are separated into four major groups by the HCA dendrogram which are spaghetti processed samples (8, 6 and 5), Semo processed wheat samples

(13, 14 and 7), unprocessed wheat flour samples (11,12 and 10) and processed wheat four sample (4, 2, 1 and 9).

The hierarchical analysis (HCA) dendrogram of the farinograph properties of the unprocessed and processed samples reported in fig. 4.6.3 reveal two distinct groups and sub-clusters.

Fig. 4.6.3 shows the clustering of the Farinography properties the 8 wheat flour samples. The classification was carried out based the similarities among 8 wheat flour samples for the 7 Farinography property variables. The HCA dendrogram showed the substitutability of wheat samples based on variance - weighted distance between cluster centers in the decreasing of 5 and 7, > 4 and 3 > 1 and 2 according to farinography properties. The wheat samples are separated into two different groups of unprocessed wheat flour samples {5(WH1), 6(WH2), 7(WH3) and 8(WH4)} and processed wheat flour samples {1(GP1), 2(GP2), 3(GP3) and 4(GP4)} by the HCA dendrogram thereby their process disparities.



Keys: 1 to 4 = processed general purpose flour, 5 to 8 = unprocessed samples.

Figure 4.6.3 Hierarchical cluster Analysis (HCA) result for farinograph properties

4.7 Pair-wise Correlation Analysis

Table 4.7.1: Correlation analysis results for proximate composition and mineral content of the processed samples

	Na	K	Ca	Mg	P	Fe	Zn	F	M	P	FB	A	C	E
Na	1.00													
K	-0.32	1.00												
Ca	0.23	0.25	1.00											
Mg	-0.13	0.72	0.46	1.00										
P	0.59	0.01	0.55	0.37	1.00									
Fe	0.32	0.44	0.86	0.65	0.70	1.00								
Zn	0.12	0.56	0.27	0.61	0.46	0.55	1.00							
F	-0.29	0.28	-0.24	0.09	-0.38	-0.19	0.24	1.00						
M	0.31	0.40	0.48	0.56	0.76	0.76	0.35	-0.48	1.00					
P	0.04	0.20	0.13	0.23	0.17	-0.02	0.42	0.44	-0.25	1.00				
FB	0.73	-0.12	0.44	-0.12	0.50	0.39	0.36	-0.28	0.15	0.32	1.00			
A	0.32	-0.70	0.18	-0.71	0.03	-0.23	-0.55	-0.38	-0.32	0.05	0.45	1.00		
C	-0.07	-0.48	-0.39	-0.28	-0.12	-0.29	0.15	0.26	-0.41	-0.15	-0.17	-0.08	1.00	
E	-0.31	0.39	0.03	0.17	-0.37	-0.05	0.19	0.92	-0.45	0.56	-0.14	-0.23	-0.04	1.00

Keys: M = moisture, FB = crude fibre, 1st P = crude protein, A = ash, E = energy, C = carbohydrate, F = fat, Na = sodium, K, potassium, Ca = calcium, Mg = magnesium, Fe = iron, Zn = zinc and 2nd P = phosphorus.

Table 4.7.2: Correlation Analysis Results for proximate composition and mineral content of the unprocessed samples

	Na	K	Ca	Mg	P	Fe	Zn	F	M	P	FB	A	C	E
Na	1.00													
K	0.28	1.00												
Ca	-0.23	0.85	1.00											
Mg	0.26	0.68	0.42	1.00										
P	0.42	-0.60	-0.90	0.00	1.00									
Fe	-0.48	0.14	0.25	0.67	0.00	1.00								
Zn	0.95	0.44	-0.01	0.18	0.14	-0.61	1.00							
F	0.39	-0.72	-0.86	-0.68	0.65	-0.70	0.30	1.00						
M	-0.06	0.10	-0.04	0.78	0.38	0.89	-0.29	-0.45	1.00					
P	0.26	-0.27	-0.26	-0.81	-0.07	-0.97	0.41	0.70	-0.95	1.00				
FB	0.33	0.99	0.80	0.77	-0.50	0.23	0.44	-0.72	0.23	-0.37	1.00			
A	-0.83	-0.66	-0.18	-0.74	-0.18	-0.01	-0.79	0.17	-0.33	0.24	-0.73	1.00		
C	-0.09	0.31	0.52	-0.48	-0.78	-0.62	0.21	-0.04	-0.87	0.68	0.18	0.23	1.00	
E	-0.05	-0.86	-0.91	-0.23	0.88	0.15	-0.32	0.59	0.36	-0.11	-0.78	0.28	-0.74	1.00

Keys: M = moisture, FB = crude fibre, 1st P = crude protein, A = ash, E = energy, C = carbohydrate, F = fat, Na = sodium, K= potassium, Ca = calcium, Mg = magnesium, Fe = iron, Zn = zinc and 2nd P = phosphorus.

The correlation matrix is important as it gives an insight regarding element interdependence in the sample analyzed. Information from correlation analysis also helps in characterizing the general profile of the samples. For the processed samples, strong correlation was observed between Energy (E) and Fat (F) and Calcium (Ca) and Iron (Fe). This result was found to disagree with that of Kibar *et al* (2019) who observed significant correlation between Ash and K (potassium) having assessed the mineral composition and morpho-physiological properties of de-hulled eikorn wheat flours.

The correlation matrix showed more element interdependence in the unprocessed samples analyzed. The information from this correlation analysis helps in characterizing the general profile of the samples. Strong correlation was observed between Fb and K, Zn and Na, Ca and K, E and P, M and Fe. The observed correlations imply that the correlating elements can go along with one another. Negative correlations were also observed amidst the unprocessed samples between E and Ca, P and Fe and C and M, which also implies that negatively correlated elements do not go along. Although negative correlations were also reported by Kibar (2019), the negatively correlating elements were observed to be different from the above report (P, Na, Ca, Mn, Co, and Cr when compared to K contents). This may be due to the difference in the nature of the samples analyzed.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

All the samples contained appreciable amounts of the various nutrients studied, no two samples had the same nutritional values. However, it was observed that samples of similar brands/identities showed close properties. Both the unprocessed and processed wheat samples were observed to contain appreciable amounts of macronutrients. Though, it was revealed that the unprocessed samples were richer in crude fat (5.59-6.92%), crude fibre (0.69-1.16%), carbohydrate (66.39-70.09%) and energy (369.71-383.95) kCal. This explains for why they are good source of calorific foods.

Also, the unprocessed samples had more appreciable amounts of micronutrients such as Na (5.91-6.82 mg/100g), K (506-572 mg/100g), Mg (46.17-66.30mg/100g) and Zn (5.19-6.43 mg/100g), while the processed samples had more P (172.40-278.32 mg/100g) and Fe (5.58-6.58 mg/100g).

The physical tests conducted revealed that processed flour samples possessed better gluten properties than the unprocessed wheat samples (sedimentation analysis). Farinography differentiated the samples into flour and non-flour samples as only the samples in the flour categories could undergo Farinography test due to lack of required rheological properties. The processed wheat flours possess higher baking properties compared to the unprocessed wheat samples. This may be due to the differences in the nature of samples as processed samples may have undergone process treatment(s) which may have enhanced it's rheological and baking properties. Application of chemometric techniques (PCA and HCA) revealed that substitutability is peculiar to either of the processed and unprocessed samples which will enable the consumers to

make informed choices, it also categorized the samples into groups such as Spaghetti processed, Semo Processed, Flour processed, more and less macro/micro nutrients rich and unprocessed and processed samples. The correlation matrix revealed element interdependence, positive correlations between energy (E) and fat (F) and iron (Fe) and calcium (Ca) among the processed samples. The unprocessed samples revealed more correlations between fiber (F) and potassium (K) and zinc (Zn) and sodium (Na). Hence, there is loss of identity to processing conditions as the signatures of the raw samples could not correlate with the processed samples.

5.2 Recommendations

Unprocessed wheat samples were observed to possess more appreciable amount of macro and micro nutrients (such as Na, Ca, K, P, Mg, Crude Fiber, Fat, Energy and so on) with lesser gluten contents. Thus, more suitable for consumption as they are gluten-friendly and conformed more to DRI (Dietary Reference Intake) specifications. From the result of the study, determination of heavy contents should be carried out to ascertain the safety of the samples. Also, analysis such as Rising and Falling numbers, Maltose/diastatic activities, Vitamin A and Alveography (flour samples only) can be carried out for further studies to add to the existing information.

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